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Journal

—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries.

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JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 1 **June, 1918** **No. 6**

EDITORIALS.

CLAY PRODUCTS AT THE CHEMICAL EXPOSITION.[1]

A survey of the exhibits at the Grand Central Palace revealed the fact that while many products of the ceramic industries, other than glass, are indispensable in the processes of chemical manufacture and research, the exhibits of these were not very numerous. There were found at the Exposition three displays of chemical porcelain, two of chemical stoneware, one of fire brick, three of laboratory refractories, one of insulating material, one of vitrified cells for acid towers, and two of enameled iron.

Chemical Porcelain.

The exhibits of chemical porcelain were those of the Coors Porcelain Company, the Guernsey Earthenware Company, and the Ohio Pottery Company. Inasmuch as no criterion for chemical porcelain can be advanced without the evidence afforded by special tests, it is not possible in a review such as this to make any mention of quality. It will suffice, therefore, to describe the three exhibits as one, especially as the same excellence in manufacture and resource is evident in each case. The improvements effected since the Exposition of last year were quite marked. All the factories are now successfully producing evap-

[1] Fourth National Exposition of Chemical Industries, Grand Central Palace, week of September 23, 1918. Reference to the Chemical Exposition in the June number of THIS JOURNAL is accounted for by the fact that the January number of Vol. 1 of the JOURNAL was issued in July of this year—the succeeding numbers being correspondingly late.

orating dishes, both flat and curved, of twenty-inch diameter, while of course the usual laboratory sizes of these, together with beakers, crucibles, casseroles and spatulas, were strongly in evidence. The mastery of the technical difficulties in the workshop is well shown by the excellent production of delicate porcelain plates with minute perforations. Some of these form the bottoms of Gooch crucibles, while others are detached. Certainly no finer specimens of workmanship are made anywhere.

Chemical Stoneware.

The two exhibits of chemical stoneware may likewise be considered together. They were by the General Ceramics Company, of Keasbey, New Jersey, and Maurice A. Knight, of East Akron, Ohio. The modern chemical stoneware is thoroughly vitrified throughout. It is said that the glaze is added for appearance only, but the smooth polished surfaces of pump plungers and valve balls is satisfactory both to the sight and touch. The structural intricacy of some of the pieces is a marvel to any observer, but to those who are versed in the especial difficulties of clay-shaping and firing, it is a veritable miracle. No problem of construction seems to baffle these potters; huge tanks and jars which would drown a goodly proportion of the Forty Thieves, condensing worms larger than any barrel, valves, spigots, traps, siphons in great variety, and all of them perfect, sonorous and acid-proof pottery. Certainly there is no need for imported wares in this department of ceramics.

The exhibit of vitrified packing for acid towers seemed to be something new. The B. Mifflin Hood Company, of Atlanta, Georgia, have produced, apparently from an auger die, a tube about two inches in diameter which contains a spiral coil. The ingenuity of the product itself is quite fascinating and, given a burned clay perfectly resistant to the acid, the piece would seem to be ideal for its purpose. A large exposed surface is combined with a minimum weight.

Refractories.

The only exhibit of fire brick was that of the Didier-March Company of Keasbey, N. J. The wares, so far as inspection might

warrant, were of excellent quality and the only regret is that other manufacturers did not exhibit. Fire brick are not, of course, an accessory special to chemical manufacture and yet, in these days of high temperatures, chemists have a very real interest in the quality of refractories.

Of more direct interest were the displays of laboratory refractories by the Carborundum Company, of Niagara Falls, New York, the Denver Fire Clay Company, of Denver, Colorado, and the Norton Company, of Worcester, Massachusetts. The first named showed especially the larger pieces, such as muffles and crucibles of carborundum bonded with clay. The Denver Fire Clay Company had an excellent display of fire clay crucibles of all sizes, and a complete line of cupels and scorifiers. The Norton Company specialized in the smaller wares, some of which were intricate in form and all were well made—crucibles and boats for analytical work, tubes for electric furnaces and pyrometers, as well as small cones and thimbles for special operations.

The Sil-O-Cel Company showed a collection of insulating material with which an interesting and effective exhibit was made. Several illustrative units were shown in which the insulating properties of the material were compared with those of fire brick and other standard materials.

THE GLASSWARE EXHIBITS AT THE EXPOSITION.

Among the ceramic exhibits, those of glassware proved to be of high interest. Since the exhibition was limited to products of direct relation to the chemical industries, the glassware exhibits represented a very small field of the entire glass industry, but this field is one of the greatest importance to the chemical industries and to those industries in which chemists and chemical laboratories are essential.

The glassware exhibits comprised three groups, namely, exhibits by glass manufacturers; exhibits by apparatus dealers, by whom the bulk of the special apparatus is made before the lamp; and exhibits by manufacturers of fused silica.

Of the glass manufacturers, the exhibit of the Corning Glass Company was the largest and most elaborate. A variety of "Pyrex" laboratory apparatus and baking dishes formed the

basis of the display. In addition, there were exhibits of "Frensel" lenses and other signaling lights in a variety of colors as well as of lenses for automobile and other headlights. One of the features of this exhibit was the glass tubing for special purposes. In this was included tubing for burettes and "lens" tubing especially for clinical thermometers. Demonstration was also made of a lamp equipped with daylight glass. Popular attention was attracted by a glass blower who demonstrated the simplicity of working glass before the lamp.

The booth of the Macbeth-Evans Glass Company was devoted to an exhibit of their well-known laboratory glassware. The exhibit was simple, well chosen, and displayed the usual excellent workmanship. One feature of the exhibit was a twenty-inch "balloon flask" (18 gallons), used in mixing fulminate compositions.

One exhibit of the Whitall Tatum Glass Co. consisted of a simple display of the long and favorably known "non sol" chemical laboratory apparatus, together with a selection of reagent bottles in a variety of forms.

Of the dealers' exhibits, that of the Emil Greiner Co. was of commanding interest. It might almost be characterized by "anything with a glass stopcock." This was not, however, the limit of the exhibit. Thermometers, intricate absorption apparatus, condensers, fractionating columns and Dewar flasks, were features.

The glass exhibit of the Central Scientific Co. comprised only a small portion of their total display. One exhibit was of interest, however, in view of the standardization of sizes and shapes of laboratory apparatus and the construction of special apparatus in accordance with the specifications of American scientists.

An industry comparatively new to America is the production of fused silica and quartz glass. The former, the opaque product, and the latter, the transparent variety, were exhibited by the Sidio Co. of America and by the Hanovia Chemical and Manufacturing Co., respectively. Although these exhibits were limited to small- and medium-sized apparatus, the excellence of workmanship indicates that real progress is being made in this new and important industry.

The Thermal Syndicate also presented an exhibit of their well-known products. This exhibit was characterized by a display of the larger sized apparatus. The features were a nitric acid condensing system, of elaborate construction, and a display of drawn tubing in ten-foot lengths and ranging up to four inches in diameter. A line of the smaller articles in transparent silica has recently been added.

Akin to the exhibits of glassware were those of enameled iron apparatus displayed by the Elyria Enameled Products Co. and by the Pfaudler Co. These exhibits deserve more than the passing mention here given them, for they represent real progress in an important field of ceramics.

The glass exhibits, as a whole, were pleasing and were creditable to the American industries. It is to be regretted that a larger number of manufacturers of glass apparatus were not represented, but the exhibits demonstrate that the necessary technical knowledge is not wanting in this line. It is reassuring to observe that the principal handicap, the lack of manual and mechanical skill, is rapidly being overcome and that a permanent American industry in scientific glassware is being surely established.

AMERICAN CERAMIC SOCIETY EXHIBIT AT THE EXPOSITION.

By C. H. Kerr.

One of the features of the Ceramic Section of the Exposition was a series of eight charts displayed upon the walls of the American Ceramic Society's booth. The interest and comment which the charts aroused at the Exposition seemed to warrant their reproduction in the JOURNAL and they are therefore presented in this issue.

Very few, even of the members of the American Ceramic Society, have any clear conception of the great magnitude of the ceramic industries, either considered as separate branches of the industry, such as fire brick, pottery, glass, cement, etc., or considered in the aggregate, and it is not at all surprising, therefore, that the general chemist or the specialist in some other branch of applied science should be quite startled to learn that the value of ceramic products is about one-third greater than the value of electrical machinery, about two and a half times

the value of the potato crop of the Nation, and more than twice the value of the petroleum products of the country.

While the charts show the approximate figures, it may be of interest to record the values, in round numbers, from which the charts were prepared. The figures are the Government official figures, taken from the Census Reports for the year 1914. Later reports are available in many instances but the World War has created such havoc with industrial statistics that the older figures are considered more reliable in showing the relative proportions of the various industries.

For the Manufactured Products the figures used in Chart No. 1 were:

Cotton goods	$701,000,000
Men's clothing	584,000,000
Chemical industries	548,000,000
Ceramic industries	447,000,000
Petroleum refining	396,000,000
Woolen goods	395,000,000
Electrical machinery	335,000,000
Blast furnace products	318,000,000

For Mineral Products the figures used in Chart No. 2 were:

Coal, soft	$493,000,000
Ceramic products	447,000,000
Iron ore	299,000,000
Petroleum	214,000,000
Coal, hard	188,000,000
Copper	153,000,000
Gold	94,000,000
Silver	40,000,000

The statistics regarding Agricultural Products (Chart No. 3) are of the greatest interest, for, after all, the United States is chiefly an agricultural nation though we are very apt to overlook that fact because the agricultural production is made up of innumerable small units rather than large, segregated groups. We do not have a farm or a group of farms to compare with the United States Steel Corporation, but we do have the grand

totals of agricultural products that dwarf even our immense totals in manufactured products. The charts showing the relative value of Ceramic Products in comparison with agricultural Products is based upon the following figures:

Corn	$1,722,000,000
Wheat	878,000,000
Hay	779,000,000
Cotton	720,000,000
Oats	499,000,000
Ceramic products	447,000,000
Potatoes	199,000,000
Tobacco	96,000,000

In all of the above statistics there are included in the ceramic industries the manufacture of clay wares of all kinds, glass, cement, enameled wares, plasters, lime, abrasive products and allied articles usually understood as being embraced within the ceramic field. To analyze the items, making up the impressive aggregate of 447 million dollars a year for the ceramic industries, would be very instructive and very surprising, but that must be left for a later issue of the JOURNAL.

The most interesting curve shown at the Society's booth was that which displayed in graphic form the growth of the American Ceramic Society (Chart No. 4). Founded in 1898 and starting its real existence in 1899, the membership shows a steady and consistent growth to somewhat over 500 in 1917. In 1918 the growth has been unusual and the membership has more than doubled. At the time of the Exposition the Society had about 1100 members enrolled. It is eminently fitting that the ceramic industry, one of the most ancient of all the manufacturing arts, should have as a clearing house for its scientific activities, an organization with the size and power of the American Ceramic Society.

Very few people, even among those engaged in ceramic work, realize the great number of ceramic products which involve the use of the various every-day raw materials, and still fewer appreciate how exceedingly vital the ceramic industry is today in supplying the products upon which other industries depend. These

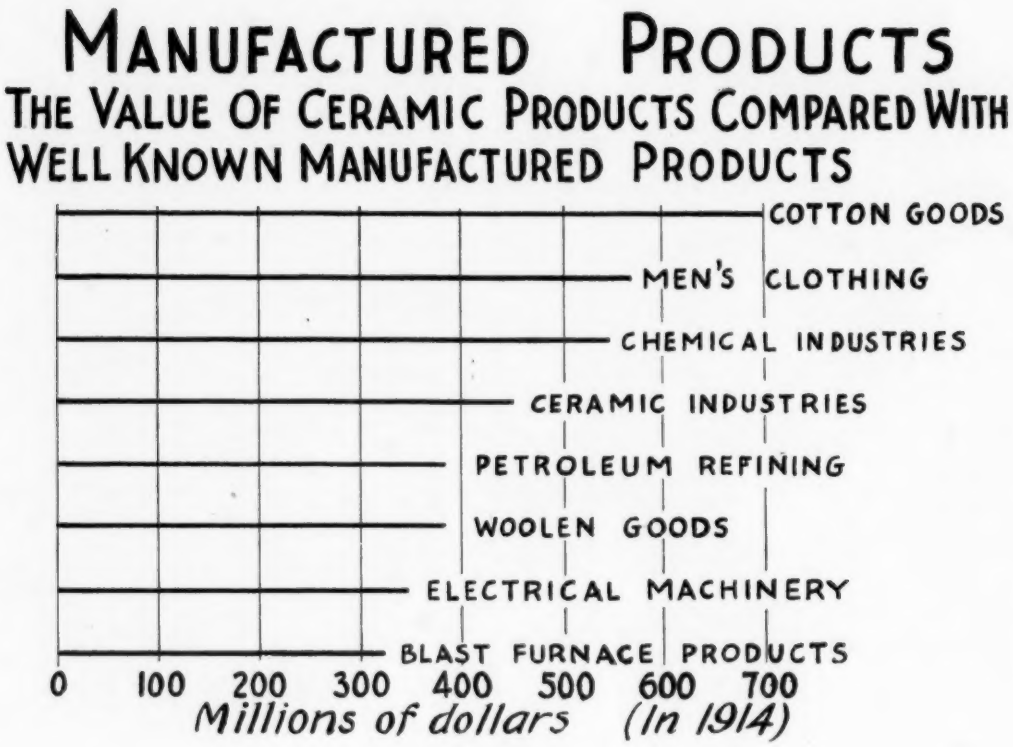
MANUFACTURED PRODUCTS
THE VALUE OF CERAMIC PRODUCTS COMPARED WITH WELL KNOWN MANUFACTURED PRODUCTS
COTTON GOODS
MEN'S CLOTHING
CHEMICAL INDUSTRIES
CERAMIC INDUSTRIES
PETROLEUM REFINING
WOOLEN GOODS
ELECTRICAL MACHINERY
BLAST FURNACE PRODUCTS
0 100 200 300 400 500 600 700
Millions of dollars (In 1914)

CHART NO. 1.

MINERAL PRODUCTS

THE VALUE OF CERAMIC PRODUCTS COMPARED WITH WELL KNOWN MINERAL PRODUCTS

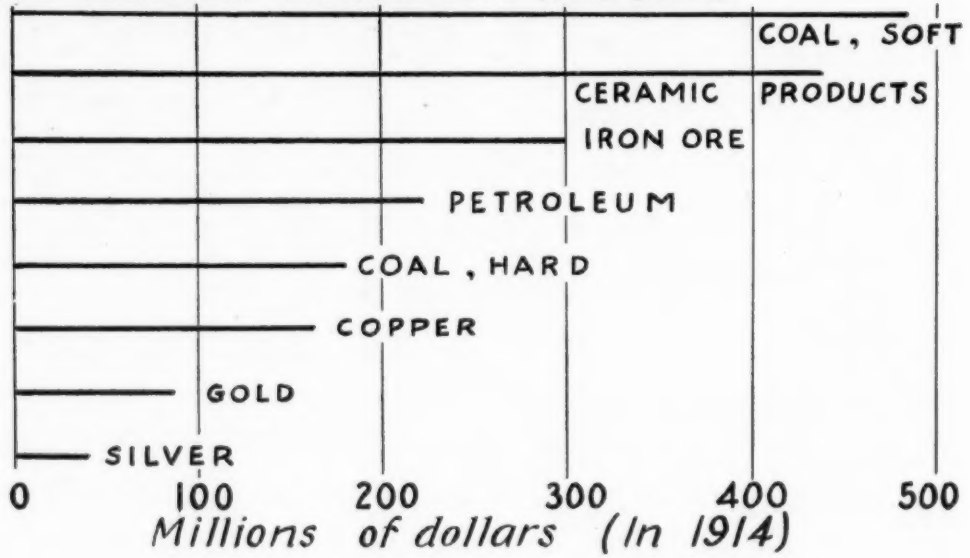

CHART No. 2.

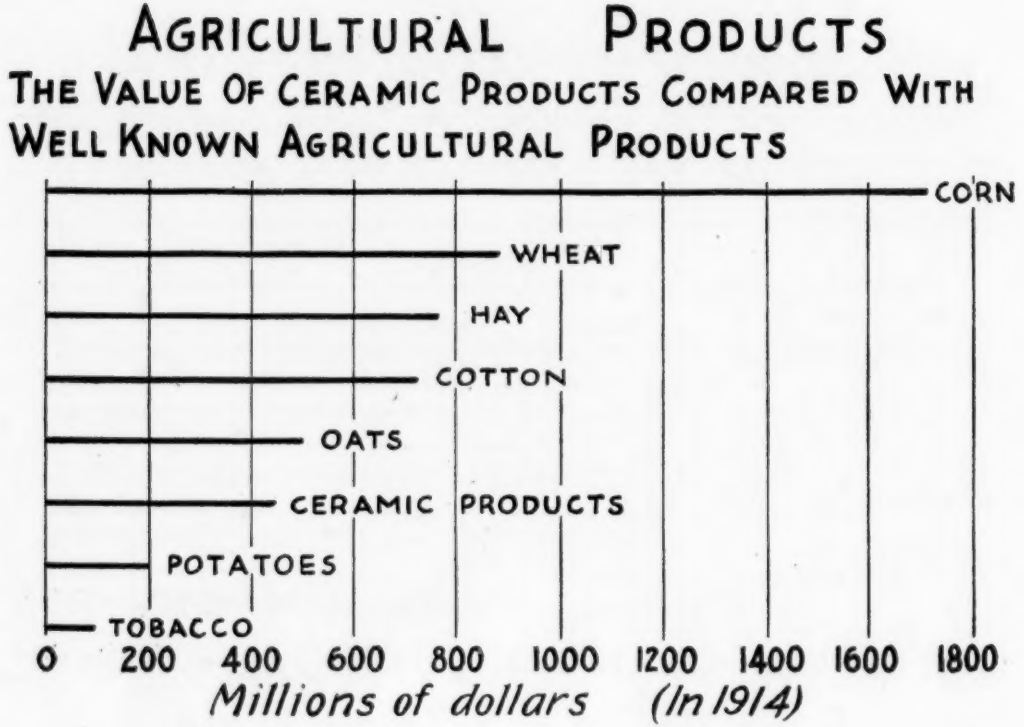
AGRICULTURAL PRODUCTS
THE VALUE OF CERAMIC PRODUCTS COMPARED WITH WELL KNOWN AGRICULTURAL PRODUCTS
CORN
WHEAT
HAY
COTTON
OATS
CERAMIC PRODUCTS
POTATOES
TOBACCO
0 200 400 600 800 1000 1200 1400 1600 1800
Millions of dollars (In 1914)

CHART NO. 3.

MEMBERSHIP GROWTH
CURVE SHOWING AMERICAN CERAMIC SOCIETY'S
GROWTH SINCE ORGANIZATION IN 1899
1200
1000
800
600
400
200
0
1900 1902 1904 1906 1908 1910 1912 1914 1916 1918

CHART NO. 4.

RAW MATERIALS
OF THE CERAMIC INDUSTRIES AND THE PRODUCTS MADE FROM THEM

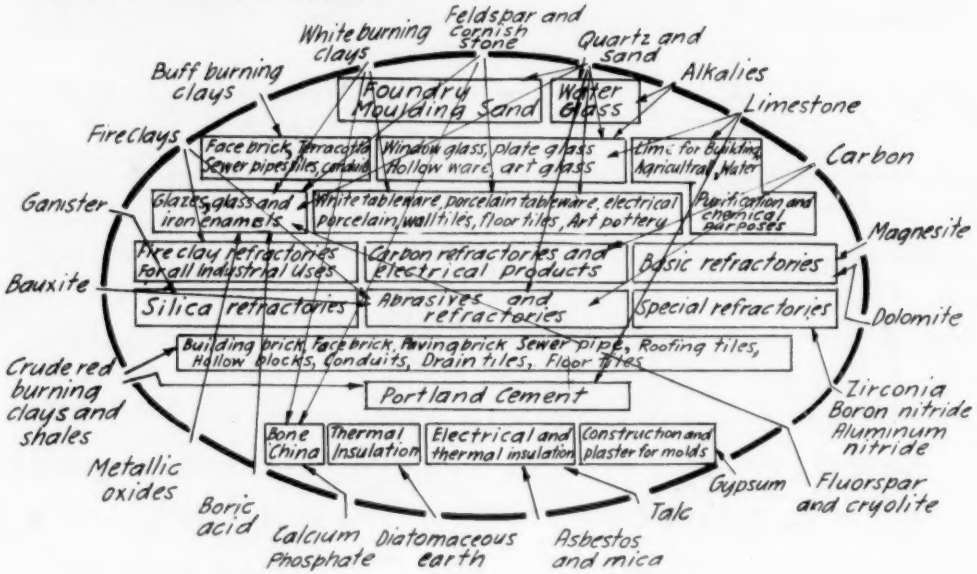

CHART NO. 5.

DEPENDENT INDUSTRIES
AND THE CERAMIC PRODUCTS UPON WHICH THEY DEPEND

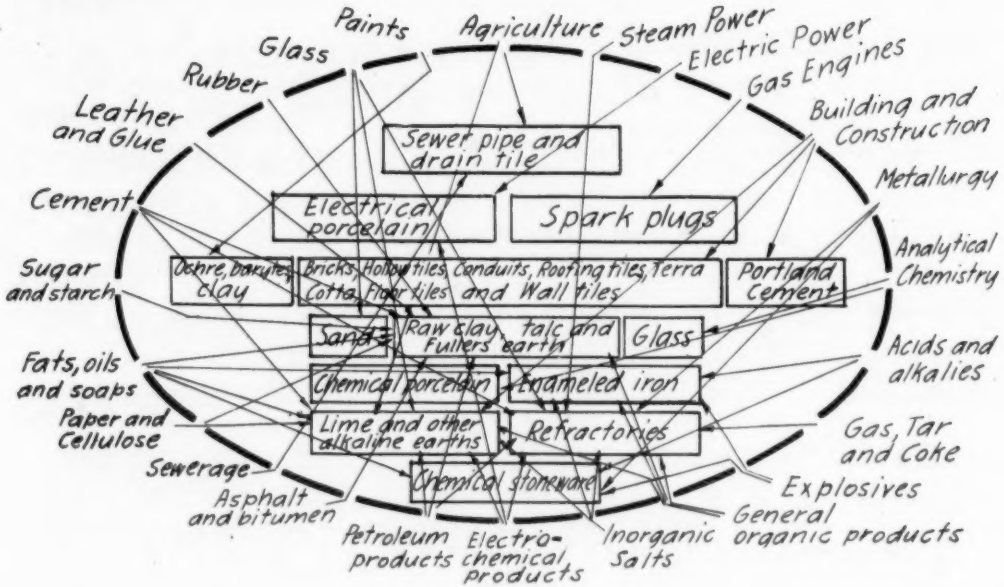

CHART NO. 6.

THE CERAMIC INDUSTRIES

A CLASSIFIED LIST OF THE BETTER KNOWN BRANCHES OF THE CERAMIC INDUSTRIES

CLAYWARES
Building brick, Face brick, Paving brick, Terra cotta, Hollow tile, Art pottery, White tableware, Porcelain tableware, Bone China, Insulation-Heat & Electrical, Cooking ware, Refractories for furnace work in all industries, Fire brick, Electrical porcelain, Chemical porcelain, Roofing tile, Floor tile, Wall tile, Glass pots & Blocks, Crucibles, Retorts, Sanitary ware, Chemical Stoneware, Sewer pipe, Drain tile, Conduits, Spark plugs

GLASS
Window glass, Plate glass, Hollow ware, Optical glass, Lenses, Chemical glassware, Water glass, Insulation, Art glass, Vault lights, Electric light bulbs, Lighting shades & reflectors

CEMENT
Portland cement, Natural cement, Slag cement

ENAMELLED WARE
Enamelled iron, Enamelled sheet steel, Enamelled insulation, Enamelled jewelry

ABRASIVES
Emery, Corundum, Sand, Garnet, Artificial abrasivies chiefly Al_2O_3 and SiC

SPECIAL REFRACTORIES
Silica brick, Quartzite brick, Magnesia products, Dolomite products, Alumina products, S.C. products, Pyrometer tubes

LIME
Limestone, Crushed rock, Quick lime, Hydrated lime, Agricultural lime, Chemical Lime

GYPSUM PRODUCTS
Wall plasters, Moulding plasters, Fertilizer materials, plaster cements

MISCELLANEOUS
Ochre, Barytes, Talc, Pumice, Rouge, Infusorial Earth, Sand, Quartz glass

CHART NO. 7.

VITAL WAR PRODUCTS
OF THE CERAMIC INDUSTRIES
A FEW OF THE PRODUCTS AND A FEW OF THE USES
NEW PRODUCTS

NEW REFRACTORIES for Destroyers and other Battle Craft
NEW SPARK PLUGS for Air plane engines. A radical development
OPTICAL GLASS for gun lights, Range finders, Binoculars, Periscopes, etc.
SPECTACLE GLASS for Spectacle lenses, Reading glasses and Simple magnifiers
SPECIAL GLASS for searchlight reflectors, Port lights, etc.
LABORATORY GLASSWARE AND PORCELAIN for control work in all industries

STAPLE PRODUCTS

FIRE BRICK for Iron and Steel works, Foundries, Glass plants, etc.
ABRASIVES for grinding optical products, Munitions, Machine parts, etc.
CHEMICAL STONEWARE for making Acids, Heavy chemicals, Explosivies, etc.
PORTLAND CEMENT for Ships, Building construction, etc.
LIME STONE AND LIME for Blast furnaces, Chemical plants and Buildings
BUILDING BRICKS AND PRODUCTS for Factory and Government construction work
ENAMELLED WARE Iron for Chemical and explosive plants; Steel for Hospital, Camp kitchen, Gallery uses, etc.

CHART NO. 8.

two relationships are shown in two of the charts (Nos. 5 and 6), which have been reproduced, but it is obvious that it has not been possible to include in such condensed form anything like a complete list, either of the raw materials and their uses, or of the other industries dependent upon ceramic products. There are, however, many very interesting points shown. We are apt to forget that the manufacture of explosives is absolutely dependent upon chemical stoneware and enameled iron with which to carry on the manufacturing processes; that gas engines are useless without spark plugs; that all metallurgical processes, especially our iron and steel industries, are dependent upon serviceable refractories; that our ability to live comfortably in houses depends upon glass for windows; that our entire city life is based on good sanitation which is made possible only by the use of ceramic products in the sewerage systems, etc. This dependency upon the ceramic industries might be shown in almost innumerable instances, but even the limited number shown in the charts is a very convincing argument of the vital need of ceramic products in our modern scheme of life.

The chart (No. 7) listing the better known branches of the ceramic industry is self-explanatory, but the one (No. 8) which tabulates a few of the vital War products calls for a little further discussion. From what has been said in connection with the other charts, it is quite plain that any list that pretends to give, with any degree of completeness, the ceramic products vital to the prosecution of the War, would necessarily be a very long and complicated one, but in order to bring to public attention a few of the most obvious or newest products the chart of Vital War Products was prepared. Ordinarily, one would not think of the refractories in the boiler room of any of our battle-craft as being of more than passing import, but this problem has been exceedingly critical and has now been satisfactorily solved. For airplane engines, a new and better spark plug was required, and it has been and is being produced. Optical glass has had national attention and everyone is familiar with the story of the American manufacturer having successfully produced the required types, qualities and quantities, so that optical glass is not a limiting feature in the National War program. Labora-

tory glassware and porcelain of American make are now available in satisfactory quantity and of good quality. And so the list could be extended to the limit of space available, or to the limit of the reader's patience, but the importance of these vital products should be indelibly impressed upon the public at large so that public opinion will see to it that in future Governmental action these all-important industries are thoroughly appreciated and their encouragement and continuance thereby guaranteed.

ORIGINAL PAPERS AND DISCUSSIONS.

THE POROSITY AND VOLUME CHANGES OF CLAY FIRE BRICK AT FURNACE TEMPERATURES.[1]

By George A. Loomis.

I. Introduction.

General Considerations Regarding the Testing of Fire Bricks.—The testing and classification of clay fire bricks is a subject which has received considerable attention in recent years, but which has not been worked out as yet—to any great degree of completeness. Failure to satisfactorily classify clay fire bricks has been due in part to the fact that clay refractories are used under such a variety of conditions. A fire brick which proves very satisfactory under certain conditions of use may not be able to fulfill the requirements for other uses. Fire bricks may be called upon to resist deformation under excessive or very light loads at high temperatures, to resist sudden changes in temperature, to withstand the intrusion of slags or glasses, to resist abrasive action, and many other equally severe conditions. Obviously, no brick could be expected to withstand all of these conditions perfectly, and it would be hardly fair to condemn a brick, for all purposes, because of its failure to withstand any one of the above conditions. However, it is generally conceded that refractoriness is a primary requisite of a fire brick for general use and the other properties, such as ability to resist abrasion or the penetration of slags, may be said to be of secondary importance. Therefore, the question of the satisfactory evaluation of refractoriness has proven the main problem in the testing and classification of clay fire bricks.

[1] By permission of the Director, Bureau of Standards.

The Measurement of Refractoriness.—In working out a satisfactory classification of fire bricks based on refractoriness, several tests for the measurement of this property have been put into more or less general use. Of these, the three which have been used the most are, first, the chemical analysis, which gives a theoretical indication of refractoriness, second, the direct determination of the so-called "softening point," and third, the determination by an actual "load test" of the ability of a fire brick to resist deformation under loads at high temperatures. These tests will be described more in detail later. Another test, as a measurement of refractoriness, but which has not been used very extensively in the case of fire bricks, is the determination of the porosity and volume changes on heating the bricks to different temperatures. A decrease in porosity and volume indicates the progress toward vitrification, and when these changes are plotted in the form of curves, the slope of the curves shows the rate of vitrification. Over-firing is then indicated by an increase in porosity and by an increase in volume as the vesicular structure accompanying over-firing is developed. A classification of fire clays, based on porosity changes, was first suggested by Purdy,[1] who set the limits for the different grades of refractories. These limits have since been found to be in error but nevertheless the idea of measuring the refractoriness of a fire brick by its changes in porosity and volume is evidently a sound one.

Temperature-Porosity Changes as an Indication of Load Carrying Ability.—The value of the porosity and volume change determinations is all the more evident when it is realized that the degree of vitrification, as shown by the changes in porosity, also represents the amount of softening which must necessarily take place in the process of vitrification. Marked softening of the mass of the brick means decreased resistance to deformation. In other words, the changes in porosity may be said to bear some relation to the resistance to deformation of a brick under load at high temperatures.

Object of Present Investigation.—From the above considerations it is evident that the determination of the porosity

[1] *U. S. Geol. Surv. Bull.*, **9**, p. 111.

and volume changes is very valuable in the study of the fundamental properties of clay fire bricks and it is only by a better knowledge of the fundamental properties that a completely satisfactory classification of fire bricks can be worked out. The present investigation was, therefore, undertaken in order to study the fundamental properties of clay fire bricks by a comparison of their changes in porosity and volume, upon heating to different temperatures, with the results of a specific load test and with the so-called "softening points." Preliminary work on this investigation was first undertaken under the direction of G. H. Brown, when connected with the Bureau of Standards. It was afterward decided to take up the investigation on a larger scale, with the coöperation of the Refractories Manufacturers' Association, which kindly furnished samples of a great number of brands of fire brick for the tests, and likewise in the interest of the American Gas Institute.

II. Description of Fire Brick Tests Already Used.

Before taking up the details of the present investigation, it is well to describe and discuss briefly the most noteworthy tests which are being used in determining the value of clay fire bricks—including the tests which have already been mentioned.

Chemical Analysis.—The chemical analysis has been used to quite an extent in the study of the fundamental properties of fire bricks and is of considerable value—not only in this regard but also in explaining the causes of failure in use. The chemical analysis serves as an indication of the refractoriness by showing the degree of purity of the clay. Clay of the purest type possible, *i. e.*, pure kaolin, has a softening temperature or so-called "melting point," of 1740° C.[1] This purest form of clay has a composition corresponding to that of the mineral kaolinite, being composed of 46.3 per cent silica, 39.8 per cent alumina and 13.9 per cent chemically combined water. In burning, this chemical water is driven off and the composition becomes 53.8 per cent silica and 46.2 per cent alumina. Clay of this composition is of comparatively rare occurrence, owing to the presence, in most

[1] *Bur. Standards, Tech. Paper,* 10.

cases, of quartz, feldspar, magnesia, lime and iron oxide, in varying amounts, and which behave as fluxes in lowering the softening point of the clay to a greater or less extent. Some attempts have been made to apply the chemical analysis directly as a measure of refractoriness, by means of formulas, but these have not been in any degree satisfactory. The determination of the amount of these fluxes by a chemical analysis may, however, be considered as an aid in determining the refractoriness of the clay, as well as serving in some measure to explain the failures of fire clay brick in use.

A classification of fire bricks based on the chemical analysis has been proposed[1]—which fixes the limit for the permissible amount of fluxes for No. 1 grade bricks. According to this classification, the total amount of fluxes must not exceed 0.22 molecular equivalents. This expression for the amount of fluxes is a part of the empirical formula commonly used in the study of ceramic bodies. This is calculated from the chemical analysis, by dividing the respective percentages by the molecular weights of the oxides, then dividing each value obtained by the value for the alumina. The values for the various fluxes are then summed up and the formula obtained is of the type $x\mathrm{SiO_2}.1\mathrm{Al_2O_3}.y\mathrm{RO}$. In this formula, determined for fire bricks, the RO equivalent must not exceed 0.22 for the No. 1 fire bricks, according to the suggested classification. In the case of the silicious clay brick (say one containing 65 per cent or more of silica) the permissible amount of fluxes is considerably less—since the fluxes are more effective upon silicious material. It is quite evident, then, that the chemical analysis is of some value in studying the causes of failure of refractories. However, there are physical tests which require less time to perform than the chemical analysis and yet which indicate much more satisfactorily the value of a fire brick in a practical manner.

Softening Point.—The determination of the so-called "softening point" is one of the most widely used tests for fire bricks and is generally considered a reliable indication of the refractoriness of the material. In determining the softening point, small tetra-

[1] *Bur. Standards Tech. Paper*, **7**.

hedra, of the size of the well-known Orton standard higher pyrometric cones, are molded from a portion of the brick, ground to pass about an eighty-mesh screen, and mixed with a gum of tragacanth solution to serve as a temporary bond. These are then set in a suitable plaque of refractory clay material beside a series of standard pyrometric cones and heated in a carbon resistance electric furnace, or a small pot furnace heated with a gas-blast burner of the Fletcher type. The "softening point" is indicated by the point at which the test cone deforms to the extent that the tip is on a level with the base or the cone has swelled excessively. Results are expressed in terms of the standard cone which has deformed to the same extent. Temperatures are not usually considered—since it is a well-known fact that the bending of the standard cones is affected by the rate of heating. However, very good comparative results are obtained by limiting the time of firing in such a test to an hour and a half or two hours.

In classifying fire bricks according to the softening point, cone 30 is usually considered the lowest value for No. 1 grade, cone 30 to 28 for No. 2 grade, and cone 28 to 26 for No. 3 grade. This classification is, no doubt, a proper one but at the same time it quite often proves misleading, in so far that the bricks with a higher softening point, cone 32 or above, sometimes fail to withstand practical conditions of load and temperature while silicious bricks, low in fluxes, with a softening point as low as cone 29, generally stand up well under heavy loads at fairly high temperatures. This is explained by the fact that silicious bricks remain quite rigid to within about 100° C of their softening points while the brick lower in silica soften through a longer range. Then, too, the softening point determination is misleading in the case of bricks made from a coarse mixture of very refractory non-plastic clay (flint clay) with an inferior bond clay. The comparatively fine grinding necessary to molding the cone brings about a more intimate mixture of the material so that the refractoriness is raised. This objectionable feature of the softening point test is sometimes overcome by using a chip of the original brick—cut on a carborundum wheel to the form and size of the standard cones. This, however, is often a difficult task and is practically impossible in the case of a very coarse friable material,

to say nothing of the variation in the results arising from not obtaining an average sample from so small a specimen.

The Load Test.—What is known as the standardized "load test" was first used by the Bureau of Standards in an investigation of clay refractories. This test has proven the most practical and surest means of determining the behavior of fire bricks in use under high loads and temperatures. The test is generally performed upon a brick of standard size and shape, placed on end in a specially designed furnace of about 20″ × 20″ × 20″ internal dimensions. The load is applied to the brick longitudinally, through a suitable opening in the top of the kiln, by means of a highly refractory column on which rests a horizontal beam. On the furnace used by the Bureau of Standards, this beam rests upon a knife edge on the column and acts as a lever fastened and adjustable at one end to keep it level, and the load is applied at the other end. On a later type of load test furnace, the beam is balanced by applying an equal load on both ends and no knife edge is used. The firing, in the case of the Bureau of Standards furnace, is done by means of eight Fletcher gas-blast burners, two on each side, and four in front. In conducting this test, the temperature is raised at a specified rate to the maximum temperature, which is held for one hour and a half. It has been found that 1350° C is the most suitable maximum temperature in testing first grade fire bricks. A load of 40 pounds per square inch has been used, although this is rather severe and one of 25 pounds per square inch is believed a fairer test for No. 1 brick.

Although there are no standard specifications for clay fire bricks as yet, one-half inch contraction in a length of nine-inch brick is considered the permissible limit for a No. 1 refractory after testing under a load of 40 pounds per square inch at 1350° C. All fire clay bricks, which have not deformed more than one-half inch in such a test, have proven very satisfactory in use under average loads at temperatures not exceeding 1350° C. Hence, there seems to be no doubt whatever of the practical value of the load test. It may be a little too severe in rejecting bricks which might prove satisfactory in use, but this fault is easily remedied by decreasing the load applied. However, there is

one objection to the load test. This is the matter of time required for its performance—since only one brick can be tested in the furnace at a time and each test requires about six hours time to perform. This is quite a serious objection when only one test kiln is available and the results on a large number of bricks are needed in a short time.

Cold Crushing Test.—This test is a rather valuable one in eliminating unsatisfactory bricks from consideration for use under loads at high temperatures. Obviously, a refractory possesses but a small part of its cold crushing strength when at a temperature of 1350° C, and if a brick has a low crushing strength when cold, it is almost certain to fail in a load test at 1350° C. It has been found that a cold crushing strength of 1000 pounds per square inch, when the brick is tested on end, is about the lowest permissible value in this regard. Needless to say, a high capacity compressive strength testing machine is necessary in performing this test.

The Ball Test.—A special form of load test, called the "ball test," has been put into use by Unger and Nesbitt, of the Carnegie Steel Company.[1] This test was devised in attempting to find a substitute for the regular load test and which could be performed on a larger number of bricks in a short time. It is claimed that the results compare favorably with those of the regular load test. The test is made by heating bricks to 1350° C for one hour in a furnace, drawing them out quickly and laying them on their side under a long lever in such a manner that a two-inch iron ball, placed on the brick under the lever, is depressed into the face as increasing pressure is applied to the lever. A certain load is applied which is gradually brought up to a maximum in about five minutes. The depression made by the ball is measured in inches. The proper load to give a depression comparable to the contraction in a regular load test was determined by experiment. There is no doubt that such a test can be made on a number of bricks in much less time than is required by the regular load test. However, there is some objection to it as a regular test for fire bricks on the ground that cooling takes place sufficiently during

[1] *Proc. Eng. Soc. Western Penn.*, **32**, No. 7.

the test to influence the results. Then, too, the portion of the brick receiving the pressure is a small part of the whole brick and the results are apt to be affected by small local defects in the structure of the brick.

The special tests of fire bricks, such as the determination of the resistance to slagging action, the resistance to impact and abrasion, and the test of ability to withstand sudden temperature changes, have little or no connection with the present investigation and need not be described here.[1,2]

III. Method of Study of Porosity and Volume Changes in Connection with Load Tests and Softening Points.

Determination of Porosity and Volume Changes.—Taking up the details of the present investigation, the porosity and volume changes of the fire bricks, for comparison with the results of load tests and softening points, were determined in the following manner: Specimens of each brand of fire brick were cut with a broad chisel into briquettes, approximately $2^1/_2 \times 1^1/_4 \times 1^1/_4$ inches in size, squared up somewhat on a grinding wheel, and suitably marked with a cobalt paint—made by mixing powdered cobalt oxide, one part, and kaolin, three parts, in water. Six briquettes were cut for each brand. A neater method of cutting the specimens would have been to use a carborundum cutting wheel. However, the method used was quicker and equally as good except for the appearance of the specimens.

The initial porosity was then determined on two of these small specimens from each brand—by first weighing the specimens to 0.10 g. after drying at 110° C, then determining the wet weight and the suspended weight after soaking in boiling water under a vacuum, represented by a 24-inch mercury column, for four hours. The per cent porosity was then determined from the usual formula

$$\frac{\text{Wet weight} - \text{dry weight}}{\text{Wet weight} - \text{suspended weight}} \times 100 = \text{per cent porosity.}$$

[1] For description of these special tests see Nesbitt and Bell, "Practical Methods for Testing Refractory Fire Bricks," *Proc. Am. Soc. Testing Materials*, **16**, 349, Part 2.

[2] Brown, "Methods of Testing Corrosive Action of Slags," *Trans. Am. Ceram. Soc.*, **18**, 277, (1916).

The volumes of the six briquettes of each brand were then accurately determined in a voluminometer of the Seger type, after soaking the specimens in oil. Duplicate determinations of each volume were made to obtain a check within 0.2 cc.

The six briquettes of each brand were then set for re-burning in a muffle in a test kiln in such a manner that a specimen for each brand could be drawn at regular intervals in the process of firing. Space was left between each set of draws and between the individual piles to allow the equal heating of all briquettes. A set of pyrometric cones and a thermocouple in a porcelain protecting tube were placed inside the muffle beside the briquettes. With this arrangement, the size of the muffle permitted the burning of 84 specimens from 14 brands at one time.

In burning the briquettes, the temperature was brought rapidly up to 250°–300° C and held for three or four hours—in order to vaporize the oil in the briquettes (without igniting and leaving carbon to influence the results by the reduction of the iron). Over night, the temperature was brought up to 800° C, this being the highest temperature that could be attained in this kiln without the use of compressed air—which was not usually available at night. From 800° C the temperature was raised rapidly to 1200° C. The temperature was then increased at the rate of 30° C per hour from 1200° to 1500° C. The draws were made at 50° intervals between 1250° and 1500° C, and were cooled slowly by covering with hot sand in a suitable container.

The deformation of the standard cones, as observed during the firing of the briquettes, was as follows:

Cone 13 down at 1350° C
Cone 15 down at 1450° C
Cone 17 down at
Cone 18 half down at } 1500° C

As usual, a cone was considered down when the tip was on a level with the base. It may be well to note here that cones, when used in a burn of this kind, are retarded slightly, owing to the cooling unavoidably given them in making the draws.

After the burning of the briquettes, the porosity of each was determined by the same method used in determining the initial porosities. It may be said in regard to the method of soaking

the specimens, that placing them in boiling water under vacuum for four hours gave more consistent results than the usual method of simply boiling in water. It was found that results could be checked exactly on the same specimen by the vacuum method, whereas, results by the boiling test alone were found to be sometimes as much as three points lower than by the vacuum method.

The final volumes of the briquettes were determined in the voluminometer in the same manner as the initial volumes—duplicate determinations on the same briquettes being checked to 0.2 cc. The volume change was computed in terms of the initial volume. The volume changes and porosities at the different temperatures were plotted in the form of curves for each brand of brick.

Method of Making Load Test.—The regular load test was made on a full-size specimen of each brand (standard size bricks used) employing a load of 40 pounds per square inch at a temperature of 1350° C for an hour and a half. The load of 40 pounds per square inch was considered best for testing first grade fire bricks at the time the tests were made, although one of 25 pounds per square inch is now generally considered as best.

The kiln used for the load test was the one which has already been described. Special care was used during these tests to keep the beam level at all times in order to prevent eccentric loading on the specimen. The usual rate of firing up to 1350° C was followed. It is given in Table 1.

TABLE 1.—SCHEDULE OF FIRING IN LOAD TEST.

Time.		Temperature.	Time.		Temperature.
Hours.	Minutes.	Degrees C.	Hours.	Minutes.	Degrees C.
..	15	200	2	45	1160
..	30	370	3	..	1200
..	45	520	3	15	1240
1	..	670	3	30	1270
1	15	800	3	45	1300
1	30	880	4	..	1320
1	45	960	4	15	1340
2	..	1020	4	30	1350
2	15	1070			
2	30	1120	1350° C held for one hour and a half.		

The thermocouple, in a porcelain protecting tube, used in measuring the temperature, was placed within one-half inch of the brick. Cones were also placed close to the brick as a check on the firing—although the couples and instruments used in this test and for the porosity determinations were calibrated against standards, from time to time, to insure accuracy in measuring the temperatures.

When the investigation was first started, the plan was to make duplicate determinations by the load test and to take the average results for comparison with the results of the porosity and volume change determinations. However, the variation in initial porosity on different specimens of the same brand of brick is sometimes quite considerable, due, in most cases, to a difference in burning in manufacture. It was therefore decided to determine the initial porosity on several of the full-size bricks of each brand, in the same manner as was done with the briquettes, and to use the brick for the load test which was found to have the nearest porosity corresponding to that of the briquettes used in the other test. By so doing, it seemed that a good comparison of the results of the load test with those of the porosity and volume change determinations could be made without making duplicate determinations by the load test on each brand. Under the circumstances, it did not seem worth while to take the time for more than one load test on each brand—considering the large number of brands tested.

Method of Determining Softening Points.—The softening-point determinations were made by grinding up portions of the brick to pass a 60-mesh screen and molding with gum of tragacanth into cones as nearly as possible the exact size of the Orton higher pyrometric cones. These were set in a plaque of suitable refractory clay material with a series of pyrometric cones—in the usual manner of determining softening points—and fired in a small pot furnace, using a Fletcher blast burner with natural gas and compressed air. The time of firing in each test was limited to an hour and a half to two hours. The softening point was determined by the point at which the cones bent in the prescribed manner and the result was expressed in terms of the corre-

sponding standard cone. Duplicate determinations of the softening point were made on two bricks of each brand. Practically all of the results agreed within one-fourth of a cone and in no case did duplicate determinations on two bricks differ more than one-half cone.

IV. Discussion of Results.

A study of the data obtained shows some interesting relationships between the porosity and volume changes and the results of the load tests. In nearly all cases of brick which successfully withstood the load test—showing a deformation of not more than 5.55 per cent—there is comparatively little volume change by either expansion or contraction up to 1425° C, while a considerable number of the brick which failed in the load test show very appreciable volume changes. Similarly, the porosity decrease in the case of brick which pass the load test is not large while many of those which failed show considerable decrease in porosity at some point below 1425° C. In many cases, over-burning of the brick of poorer grade is distinctly evident from the volume and porosity changes. The sudden and more or less pronounced expansion at the lower temperatures of firing, in the case of some of the bricks, is certain evidence of over-burning which was confirmed by the results in the load tests. An abrupt increase in porosity at the same or at a slightly higher temperature is usually noted in such cases. Invariably, such brick failed in the load test. Bricks which show either marked volume change or a considerable decrease in porosity also failed to withstand the load test.

It would seem, then, that the volume and porosity changes of clay fire brick might serve in some measure as a criterion of their ability to withstand load at high temperatures. A careful study of the following data for various burning temperatures shows some interesting points in this connection:

1350° C: Of 26 brick which passed in the load test, 23 show not more than 1 per cent volume change, 3 show more than 1 per cent. Of 35 brick failing in the load test (showing more then 5.55 per cent deformation), 16 show not more than 1 per cent volume change and 19 more than 1 per cent.

Of 26 brick passing the load test, 25 show not more than 3 per cent porosity decrease, 1 more than 3 per cent. Of 35 failing in the load tests, 13 show not over 3 per cent and 22 more than 3 per cent porosity decrease.

Combining these criteria, 33 of 61 brick show not more than 1 per cent volume change nor more than 3 per cent porosity decrease—23 of these passing and 10 failing the load test. Of those showing more than 1 per cent volume change or more than 3 per cent decrease in porosity, 3 passed and 25 failed the load tests.

1400° C: Not over 2 per cent volume change was shown by 23 brick which passed and 15 which failed in the load test. Of 23 brick which showed over 2 per cent volume change, 3 passed and 20 failed in the load tests.

Of 36 brick showing not over 5 per cent decrease in porosity, 22 passed and 14 failed in the load test. Of 25 showing more than 5 per cent porosity decrease, 3 passed and 22 failed in the load test.

Of the brick which showed neither a volume change greater than 2 per cent nor a porosity decrease exceeding 5 per cent, 22 passed and 10 failed in the load test.

On the basis of a volume change not exceeding 3 per cent at 1400° C (amounting to about 1 per cent linear expansion or contraction), 24 passed and 19 failed in the load test. Of those exceeding 3 per cent volume change, 2 passed and 16 failed the load tests.

Excluding brick which showed more than 3 per cent volume change or 5 per cent porosity decrease, 22 passed and 10 failed in the load test. Of those which showed either more than 3 per cent volume change or 5 per cent porosity decrease, 4 passed and 25 failed in the load test.

1425° C: Of 38 brick showing not more than 2 per cent volume change, 23 passed and 15 failed the load test. Of those showing more than 2 per cent volume change, 3 passed and 20 failed the load tests.

Of 34 brick showing not over 5 per cent porosity change, 21 passed and 13 failed the load test. Of those showing more than

5 per cent porosity decrease, 6 passed and 21 failed in the load tests.

Of the brick which neither changed more than 2 per cent in volume nor 5 per cent in porosity, 20 passed and 7 failed in the load test. Of those changing more than 2 per cent in volume or 5 per eent in porosity, 6 passed and 28 failed in the load test.

It is apparent that the porosity and volume changes of clay fire brick, when burned at some temperature between 1350° and 1425° C, offers, in some degree, a criterion of their ability to withstand the load test. The bricks passed or rejected by the suggested limitations in porosity or volume changes at the various temperatures are nearly the same in all cases. This consistency indicates that the apparent relation between these changes and the ability of bricks to withstand the load test is more than a chance one and that certain limitations in this respect might well be used as a means of rejection of a large number of bricks which would undoubtedly fail in that test. The use of either porosity or volume changes alone offers a fairly satisfactory criterion. Taken together, the result is even better—since a number of brick failing in the load test can pass one or the other specification as regards volume or porosity changes but few of the better bricks fail to pass both.

For practical purposes, a temperature should be selected which is not too high to be readily attained under ordinary conditions, but high enough that the limitations in porosity or volume changes need not be too close to be easily measured. The use of linear measurements of expansion or contraction rather than volume measurements, would be extremely desirable from the standpoint of simplicity. From the results here obtained, a temperature of 1400° C would seem most satisfactory since the permissible limit in volume change may be made as high as 3 per cent—this practically amounting to a linear contraction or expansion of 1 per cent. A limit of 5 per cent in porosity decrease is also quite satisfactory. At this temperature also the effects of over-burning in the inferior materials is likely to become sufficiently evident to be readily detected. Nearly all of the bricks which successfully withstand the load test could meet these specifications while a majority of those that fail under load would be elim-

inated by excessive volume or porosity change, or both. It may be noted that in all cases brick which showed a porosity decrease exceeding 5 per cent, or a volume change exceeding 3 per cent, showed a deformation in excess of 5 per cent in the load test—close to the permissible limit.

In the case of clay fire brick, an increase in volume is especially significant, as it represents evidence showing that over-firing has taken place—due to a comparatively high content of fluxes. It is important, therefore, that the permissible expansion be rigidly limited to a definite value which must be lower than that for the allowable contraction.

In the case of some of the brick failing in the load test—due to failure of the bond—there were no marked changes in either volume or porosity. Generally, such brick have a very low, cold crushing strength—as in the case of one brick which failed under a load of 364 lbs. per square inch and another which crushed at 385 lbs. per square inch. The minimum allowable cold crushing strength for a No. 1 refractory is generally placed at 1000 lbs. per sq. inch.

There is apparently little relation between the softening points and the volume and porosity changes or with the results of the load test. Some of the brick, showing high softening temperatures, failed to withstand this test while several which softened below cone 31 showed less than 5 per cent deformation. The latter were probably silicious in character, containing more than 65 per cent SiO_2. In some cases where brick failed, the high softening point is probably due to the intimate mixture of the inferior bonding clay with the more refractory flint clay in the preparation of the test cones—the material being ground to 60-mesh size. In all cases, brick showing softening points lower than cone 28 failed completely in the load test. It may be considered then that cone 28 should be the minimum softening temperature of a No. 1 refractory of either silicious or non-silicious character.

In Fig. 1 are shown porosity and volume change curves for six bricks of different characteristics which successfully withstood the load test. These have been selected as fairly typical of the

bricks of this class. Contraction is indicated by negative volume change, expansion by positive values.

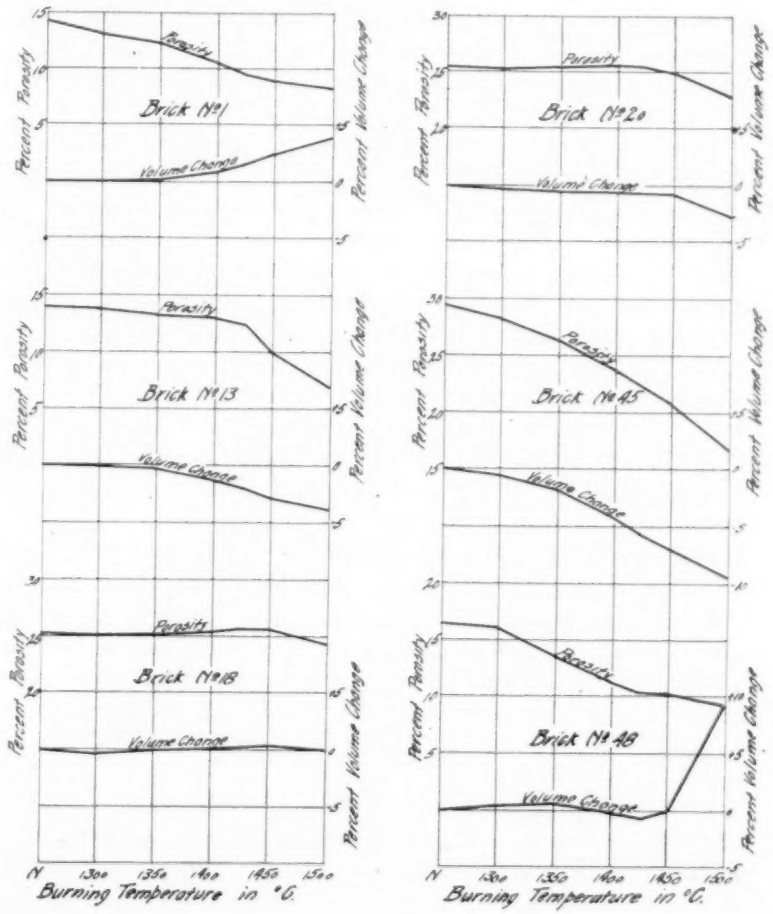

FIG. 1.

In the curves for brick No. 1 a gradual expansion, accompanied by a gradual decrease in porosity, is noted. The low initial porosity is characteristic of the stiff-mud brick. The gradual expansion would seem to indicate a silicious character, but this

seems unlikely in view of the high softening point, cone 32. The result of the load test was a deformation of 4.47 per cent.

Brick No. 13 shows gradual contraction beyond 1350° C—accompanied by an increased rate of porosity decrease above 1425° C. This is also a stiff-mud brick with low initial porosity and a softening point of cone $31^{3}/_{4}$. The deformation in the load test was slight, only 1.06 per cent.

Brick No. 18, made by dry pressing, is apparently silicious in character—as indicated by the slight expansion on heating and the very slight porosity changes. The slight deformation in the load test, 0.11 per cent, and the softening temperature of cone $29^{1}/_{4}$, which is too low for a non-silicious material of this quality, also indicate a high silica content. The chemical analysis shows 80.56 per cent SiO_2 and a very low content of fluxes.

Brick No. 20, hand made from a mixture of flint and plastic clays, represents a high clay brick of excellent refractoriness. The porosity curve shows the characteristics of flint clays—in the slight changes up to 1425° C. No appreciable volume change occurs below 1450° C. The softening point was cone 32 and the deformation in the load test was only 1.62 per cent.

Brick No. 45 showed a deformation of 5.33 per cent in the load test—close to the allowable limit. The large amounts of contraction and porosity decrease would render the quality of this brick doubtful in the light of the suggested specifications in this respect. The regularity of the porosity decrease and contraction indicates a gradual and continuous fluxing action but with no indication of over-burning. The softening point of this material was cone 31.

Brick No. 48 shows a rapid porosity decrease above 1300° C. The volume change is slight up to 1425° C, where over-burning is indicated by the subsequent expansion. While the deformation in the load test (4.47 per cent) fell within the limit, the porosity change exceeds 5 per cent. The ability of this brick to withstand a long-continued heat treatment at high temperatures would be somewhat doubtful—due to its over-burning tendencies. The softening point was at cone $31^{1}/_{4}$.

In Fig. 2 are shown curves for some of the brick which failed to pass the load tests. Brick No. 26, with a deformation of 12.6

per cent, shows a gradual expansion above 1300° C, becoming rapid above 1400° C. The increase in porosity at 1425° C indicates that this is probably due to the gradual development of a vesicular structure by over-burning.

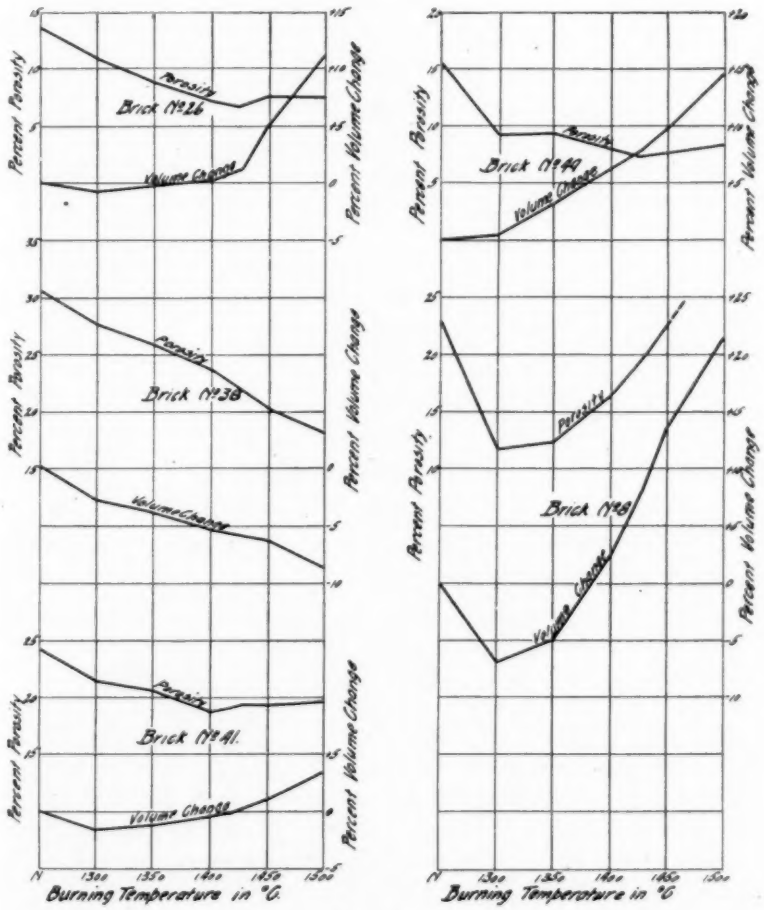

FIG. 2.

Brick No. 38 shows marked but gradual fluxing in its rapid decrease in both porosity and volume. It is evidently not of silicious character and of only fair refractoriness. The softening

point at cone $30^1/_4$ and the deformation in the load test (10.4 per cent) confirms this.

Brick No. 41 begins to over-burn at 1300° C, as indicated by the expansion and by the break in the porosity curve at a somewhat higher temperature. The high softening point, cone $33^1/_2$, is apparently the result of the fine grinding of the material in preparing the test cones. The failure to pass the load test was probably due to an inferior bond clay in the mixture.

Brick No. 49, also made from a mixture of clays, shows over-burning by the expansion above 1300° C and the break in the porosity curve. The softening point is at cone $31^1/_4$ and the deformation in the load test was 9.37 per cent.

Brick No. 8 is typical of a number of those which failed completely in the load test. It has a low softening point (cone $27^1/_2$) and shows marked over-burning above 1300° C, as indicated by the rapid expansion and by the increase in porosity. The decided contraction and decrease in porosity when burned at 1300° C indicate a low temperature of burning in manufacture.

V. Suggestions for the Practical Application of Porosity and Volume Changes of Clay Fire Brick.

The determination of the porosity or volume changes of clay fire brick, burned at some temperature between 1350° C and 1425° C, affords a means of classifying a certain proportion of brick which would fail in the load test. The limit of 3 per cent in volume change (or 1 per cent in linear contraction or expansion) and 5 per cent decrease in porosity at 1400° C, does not eliminate all brick which cannot withstand such a test, but materially reduces the number which would require such testing. In this respect, it would be of considerable advantage in comparing the quality of a large number of samples—since the porosity and volume changes may be determined readily from a single burn in a test kiln. To make standard load tests on a number of brick requires considerable time and expense even if several furnaces are available.

The determination of volume changes is comparatively simple when porosities are also determined—since the difference in the wet and suspended weights of the specimens affords a measure-

ment of volume practically as accurate as determinations by means of the voluminometer.

The porosity and volume changes in burning may also be used advantageously as a means of determining the uniformity of quality of a lot of bricks of a single brand. The behavior of such bricks should be fairly uniform if they are of equal quality. Such applications of these measurements would be of value to the manufacturer as well as to the consumer.

A study of the curves for porosity and volume changes for a series of burning temperatures offers a means of determining, in some measure, the causes of failure of certain brick in the load test. If this is due to under-burning, resulting in excessive deformation under load—due to shrinkage—a considerable decrease in porosity and volume will be found at the lower burning temperatures. Clays of inferior quality, which over-burn, will be detected by a marked expansion at some point in the burning, usually accompanied at a somewhat higher temperature by an increase in porosity—due to the development of a vesicular structure. Clays high in fluxes, but which do not readily develop a vesicular structure, show gradual but marked contraction and decrease in porosity as the burning temperature is increased.

VI. Summary.

1. Brick which are capable of withstanding a pressure of 40 lbs. per square inch at 1350° C generally show slight changes in volume or in porosity when burned at temperatures up to 1425° C.

2. The greater number of the brick which failed to pass the load tests show rather marked changes in volume or in porosity at some temperature below 1425° C.

3. Brick which show distinct over-burning by pronounced expansion at temperatures below 1400° C invariably fail in the load test. The adoption of a definite limit for the permissible expansion within the given temperature range is particularly important for detecting inferior clay refractories. The limit for expansion should be lower than that for allowable contraction.

4. The changes in volume and in porosity of brick burned at some temperature between 1350° C and 1425° C serves, in a measure, as a criterion of their ability to pass the load test.

5. Most of the brick which show a porosity decrease not exceeding 5 per cent and a volume change not exceeding 3 per cent (approximately 1 per cent in linear dimensions), when burned at 1400° C will pass the load test.

6. Brick which show a decrease in porosity exceeding 5 per cent or an expansion or contraction in excess of 3 per cent by volume (1 per cent in length) at 1400° C failed to pass the load test in nearly all cases.

7. The use of limiting porosity and volume changes for clay fire brick burned at 1400° C would serve as a means of eliminating from consideration a large number of brick which fail in the load tests.

8. Brick which fail in the load test—due to failure in the bond—may not show marked changes in volume or porosity in burning but often show very low cold crushing strength.

9. No definite relationship seems to exist between the softening point of a fire brick and its ability to withstand load at high temperatures. However, all brick which softened below cone 28, whether silicious in character or not, failed completely in the load test. It seems advisable, then, to specify cone 28 as the minimum softening point for any clay fire brick. It is probable that brick containing less than 65 per cent SiO_2 should have a minimum softening point of cone 31.

In conclusion, the writer desires to express his appreciation of the assistance rendered by Mr. D. W. Ross in conducting the load tests of this investigation and to thank Mr. A. V. Bleininger for valuable suggestions in carrying out the work.

THE CALCULATION OF THE "RATIONAL ANALYSIS" OF CLAYS.

By Henry S. Washington.

Introduction.

There are comparatively few clays used in the ceramic industries which consist of pure kaolin, $Al_2O_3.2SiO_2.2H_2O$, or of those other closely similar hydrated aluminum silicates, which, collectively, have been called "clay substance" and by other similar names.[1] By far the greater number of clays contain other minerals as well; of these, the most important are quartz, feldspars, and micas. The two most common micas, the purely potassic muscovite and the ferro-magnesian biotite, are, however, not present in large amount in any but a few clays that are of commercial value, particularly for the finer ceramic purposes. Besides the above-named minerals, numerous other minerals have been observed in clays,[2] but these, almost without exception, are present in such small amounts in clays of any notable commercial value that they may be regarded as practically negligible.

Only four minerals, then, kaolin, quartz, feldspar, and mica, need be considered for the present. As each of these influences the "burning" and other properties of a clay and of the finished products, it is of importance to be able to estimate the relative amounts of each present in any given clay, so that the proper mixtures may be made or the proper temperature and other conditions may be controlled. The ceramist endeavors to accomplish this by what is known technically as the "rational" analysis.[3]

[1] K. Langenbeck, "The Chemistry of Pottery," Easton, **1895,** p. 11; J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," London, **1913,** p. 656; H. Ries, "Clays, Their Occurrence, Properties and Uses," New York, **1914,** p. 5.

[2] Mellor, "Quant. Anal.," p. 656; Ries, "Clays," pp. 56ff.

[3] As will be seen, I am quite in agreement with Mellor ("Clay and Pottery Industries," **1914,** I, p. 109, in his low opinion of the "rational" analysis and in his view that the adjective is misapplied.

The "rational" analysis aims at the estimation of the amounts of the different minerals that are present in a clay; the object of the "ultimate" analysis is to determine the amounts of the chemical constituents of the clay, quite irrespective of the mineral forms in which they may occur.

The "rational" analysis seeks to attain its object by treatment of the clay with various strong reagents, particularly hydrochloric and sulphuric acids and alkali hydroxides. The methods and details of manipulation differ somewhat,[1] and the results of the ultimate analyses are frequently used in conjunction with the data so obtained.

Following Mellor, the essential features of the "rational" analysis are: digestion of the clay with hot, concentrated sulphuric acid and subsequent washing alternately with alkali and hydrochloric acid, to remove the "clay substance;" this may be preceded by treatment with hot alkali to remove soluble silica, or with dilute hydrochloric acid, to remove the carbonates; and it may be followed by evaporation of the residue with hydrofluoric acid to remove quartz and other silica, and determination of the alumina, which is used as a basis for the calculation of the feldspar present.

There is great divergence of opinion, even among ceramists themselves,[2] as to the value of the "rational" analysis, a crude form of which was in use as early as the end of the eighteenth century.[3] It is unnecessary, within the scope of this paper, to discuss the details of the various modifications which "rational" analysis has assumed, and it must suffice to point out a few general truths that are applicable to the methods in all forms of "rational" analysis, and that are self-evident to the mineralogist or to the chemist who has had experience in the quantitative chemical analysis of silicates.

[1] For descriptions of the methods of "rational" analysis of clays see H. A. Seger, "Collected Works," Easton, Penna., **1902,** I, p. 50; Binns, Zimmer and Orton, *Trans. Am. Ceram. Soc.*, **2,** 221 (1900); H. Bollenbach, *Sprechsaal*, **1908,** No. 25, p. 340; No. 26, p. 351; Mellor, "Quant. Anal.," **1913,** p. 658; Ries, "Clays," **1914,** p. 75; A. S. Watts, *Bur. Mines Bull.*, **53,** 38 (1913).

[2] For some discussions of the value of "rational" analyses, see C. F. Binns, *Trans. Am. Ceram. Soc.*, **8,** 198 (1906); R. C. Purdy, *Ibid.*, **14,** 359 (1912); J. W. Mellor, "Clay and Pottery Industries," **1914,** p. 109.

[3] Mellor, "Quant. Anal.," p. 657.

Some, though comparatively few, researches have been made into the action of the various reagents used in "rational" analysis on the different minerals present in clays, particularly kaolin,[1] quartz, feldspar, and mica. These researches have neglected to take into account all of the factors that may have a great controlling influence on the ultimate effect of the reagents.

Foremost among these factors is the chemical composition of the mineral treated. The "feldspar" in a clay may be the potassic orthoclase, which is, other conditions being alike, only slightly attacked by strong acids. It may be one of the series of the plagioclases, or soda-lime feldspars, which in solubility relations vary continuously from the purely sodic albite, which is about as insoluble as orthoclase, through the sodi-calcic labradorite, which is somewhat soluble, to the purely calcic anorthite, which is readily soluble, even in rather weak acid. The different micas, also, vary in solubility, muscovite being attacked with some difficulty, while biotite is much more readily dissolved, the different varieties of this also varying in their behavior. Kaolin itself varies in its resistance to reagents, both acid and alkali,[2] with its origin, the kind and concentration of acid, and other conditions. The different varieties of silica which may be present in clays vary in their resistance to different reagents.

Another important factor is the size of grain, which influences very much the extent and the rapidity of attack or solution of a mineral by reagents or solvents. It is well known that the smaller the particles of a substance the more rapidly it dissolves, the relation depending on the truth that the area of a sphere diminishes as the square, and the volume or mass as the cube, of the radius. The difference in rate of solution with size of grain is accentuated, or the solution-rate gradient becomes steeper, particularly with ordinarily "insoluble" minerals, when the dimensions of the particles are of the orders of magnitude of those

[1] This term is used here to designate any of the clay-like, hydrated, aluminum silicates, whether crystallized like kaolinite, or "amorphous" like clay substance. It is the general group name used by mineralogists.

[2] See H. Stremme, in Doelter, "Handbuch der Mineralchemie," 1914, II, p. 77.

that make clays.[1] Mellor[2] has made some experiments along this line on feldspars and micas, with instructive results, as he adopted for his work the treatment used in "rational" analysis. On treatment of a sanidine (orthoclase) with sulphuric acid, for example, he found that only 0.34 per cent was dissolved if the average diameter of grain was 0.165 mm., while 15.64 per cent was dissolved if the average diameter was 0.032 mm.[3]

Other factors that will seriously influence the results are: the concentration of the reagent, the length of time during which the material is treated, the temperature at which the mixture is treated, and whether this is attained suddenly or gradually or is uniform or varied during the treatment. All of these conditions, and some others, will affect, and generally affect very seriously, the results that are obtained by "rational" analysis.

The various factors enumerated have such an important bearing on the operation of all the modifications of "rational" analysis that its methods are scarcely susceptible of yielding satisfactory duplicate results, except by chance, and even satisfactory duplication of results cannot be regarded as evidence, still less as proof, of their correctness, when the methods are seriously faulty. One can only consider, then, that the methods of "rational" analysis can furnish little but approximate and uncertain, and probably more or less erroneous and misleading, information. It would appear, therefore, that such an analysis is anything but rational.[4] It would be better to call such an analysis "modal," as it aims at determining the "mode" or mineral composition.

It must furthermore, however regretfully, be pointed out that many writers on, and workers in, ceramics have failed to utilize even the unsatisfactory data of the "rational" analysis with proper understanding of the chemical and mineralogical principles that are involved. Examples of this are numerous in the literature,

[1] It is largely because of this that one must be cautious in applying to clays the data on solubility given in Dana and other text-books; these are rough and are based on particles of much larger size.

[2] Mellor, "Quant. Anal.," p. 662.

[3] See also Langenbeck, "The Chemistry of Pottery," p. 9.

[4] Cf. Mellor, "Quant. Anal.," p. 674; "Clay and Pottery Industries," p. 110.

and some will be given later, but it may be said here that the application of scientific methods to practical ceramics demands a greater appreciation and understanding of scientific methods than seems now to obtain.

In contrast with the uncertainties of the "rational" analysis, the "ultimate," or what is better called the "chemical," analysis can be carried out with ease and great accuracy by the usual methods for the analysis of silicate rocks.[1] For a clay the usual chemical analysis takes little more time than the rational. Its results, stated in terms of the constituent oxides, may be readily reduced to mineralogical terms by a simple process of calculation that is well known to students of igneous rocks and that will yield information as to the mineral composition of a clay that is far more accurate and certain than the information furnished by the usual "rational" analysis, even when the latter is properly executed and the calculations are correctly made.

It is the chief purpose of this paper to suggest to ceramists this simple and expeditious method of calculating the mineral composition of a mixture of silicates from the chemical analysis, and to describe a modification adapted to the mineralogical characters of clays. The method suggested and to be described is only a special application of the principles and methods of calculation of the so-called "norm" of igneous rocks.[2] The "norm" is the expression of the chemical composition in terms of certain mineral molecules which are assumed to be, and which, in the great majority of cases, are, actually present in the rock. The actual *mineralogical* composition of a rock is called the "mode," while the rock is classified by the standard *molecular*

[1] W. F. Hillebrand, "The Analysis of Silicate and Carbonate Rocks," *U. S. Geol. Surv. Bull.*, **422** (1916); J. W. Mellor, "Quantitative Inorganic Analysis," **1913**; H. S. Washington, "Manual of the Chemical Analysis of Rocks," **1918**.

[2] Cross, Iddings, Pirsson and Washington, *J. Geol.*, **10**, 604, 642; also "Quantitative Classification of Igneous Rocks," Chicago, **1903**, pp. 147, 186; G. I. Finlay, "Introduction to the Study of Igneous Rocks," New York, **1913**, pp. 150, 154; J. P. Iddings, "Igneous Rocks," New York, **1909**, I, pp. 419, 433; H. S. Washington, "Chemical Anyalysis of Igneous Rocks," U. S. Geol. Surv., *Prof. Paper*, **99**, 1151–1182 (1917): also separate publication of this portion of *Prof. Paper*, **99**, p. 1918.

composition expressed in the "norm." With clays, in general, the problem and the calculation are much simpler than they are with rocks—since in most clays we have to consider and deal with only three essential minerals, namely, kaolin, quartz, and the feldspars; the micas are of secondary importance and will be taken into account later.

Some of the ideas of the method and some of the data of the calculation have been suggested and used to a limited extent by various writers,[1] and in an interesting paper Staley[2] has suggested the application of the principles of the norm to the calculation of ceramic mixtures, especially to glazes and enamel mixtures.

Stated briefly, the principle consists in utilizing our knowledge of the various molecules which the constituent oxides are capable of forming, and which they actually do form, and in considering the minerals present in the rock or clay as made up of such molecules, the actual minerals into which the molecules combine being dependent on the conditions and being in accordance with our knowledge of the occurrence of minerals in rocks.

In igneous rocks we are dealing with solidified solutions, during whose solidification and consequent crystallization as a mixture of definite minerals, certain laws of so-called "affinity" of the various basic oxides for silica and alumina come into play and determine the actual mineral composition—which may vary somewhat with the varying conditions that obtain during the solidification.

In clays and similar bodies of secondary origin, on the other hand, we are dealing with the remnants of the alteration, decomposition, and disintegration of these minerals. In these remnants we see not only the effects of the laws just mentioned, but also the action of others which seem to pertain especially to those phases of metamorphism that are known as "alteration" and "weathering," among the products of which are the clays of the potter.

[1] *E. g.*, E. R. Buckley, "Report on the Clays of Wisconsin," p. 1901; C. F. Binns, *Trans. Am. Ceram. Soc.*, **8**, 203 (1906); J. W. Mellor, "Quant. Anal.," pp. 660, 671, 674.

[2] H. F. Staley, *Trans. Am. Ceram. Soc.*, **13**, 126 (1911); Prof. Staley's suggestion only came to my attention when the present paper had been almost completed.

Let us now consider the example which especially interests us here, and to which we shall confine our attention henceforth: a clay, whether a kaolin, or a porcelain-, ball-, fire-, brick-, stoneware-clay, or other variety.

The chemical ("ultimate") composition of this is expressed as made up of silica (SiO_2), alumina (Al_2O_3), ferric oxide[1] (Fe_2O_3), magnesia (MgO), lime (CaO), potash (K_2O), soda (Na_2O), water (H_2O), with sometimes small but possibly notable amounts of titanium dioxide (TiO_2) and carbon dioxide (CO_2), and very small and negligible amounts of other constituents.

In mineralogical or "modal"[2] composition, all clays are essentially mixtures of kaolin = Kl, $Al_2O_3.2SiO_2.2H_2O$,[3] quartz = Q, and the feldspars, orthoclase = Or ($K_2O.Al_2O_3.6SiO_2$), albite = Ab ($Na_2O.Al_2O_3.6SiO_2$), and anorthite = An ($CaO.Al_2O_3.2SiO_2$); and, in some clays, small amounts of muscovite = Mu ($K_2O.2H_2O.3Al_2O_3.6SiO_2$). For the moment, the presence of muscovite and of other minerals will be disregarded, so that the principles may be more readily explained.

The problem before us then is to express the chemical composition of a clay in terms of the minerals kaolinite, quartz and the three feldspars. Their formulas represent the *molecular* composition; thus, kaolinite is made up of one molecule of alumina (Al_2O_3), two of silica (SiO_2), and two of water (H_2O). From the analysis of a mineral the formula is found by reducing the constituents to molecules and finding the ratios of these. We analyse a pure kaolinite and, neglecting the unavoidable slight impurities,

[1] Ferrous oxide (FeO) is not considered here, as it is seldom determined in clay analyses and may be regarded as oxidized to ferric oxide during the burning.

[2] This term, used to express the actual quantitative mineral composition of a rock, is borrowed from the nomenclature of the Quantitative System of classification of igneous rocks. It corresponds to the "rational" composition of the ceramist, but it must be remarked that the chemical composition is just as rational as the mineralogical.

[3] This, the well-established formula of kaolinite, may be taken as that of the clay substance, which is represented by such minerals as kaolinite, halloysite, newtonite, cimolite, and other similar substances of more or less doubtful and possibly heterogeneous composition. They represent the aluminous molecule corresponding to the magnesian serpentine, and differ chemically only in the amount of water content.

we divide the percentage amount of alumina by its molecular weight (102), that of silica by its molecular weight (60) and that of water by its molecular weight (18). It is found that the quotients, which represent the relative amounts of the molecules, are in the ratio 1 : 2 : 2, and consequently we write the formula of kaolinite as $Al_2O_3.2SiO_2.2H_2O$. The molecular weight of kaolinite is thus 102 + 120 + 36 = 258; that of quartz is 60, that of orthoclase is 556, that of albite is 524, and that of anorthite is 278.

We have then an analysis of a clay composed "modally," as stated above, and to find its mineral composition from these chemical data we reduce the percentage amounts of the various constituents to molecules by dividing each by its molecular weight; in this case SiO_2 by 60, Al_2O_3 by 102, H_2O by 18, K_2O by 94, Na_2O by 62, and CaO by 56.

Studying the formulas of the minerals under consideration, we see that in each of the feldspars one molecule of K_2O, Na_2O, or CaO is present; Al_2O_3 is present in the three feldspars, and in each in equal ratio (1 : 1) with the K_2O, Na_2O, or CaO, and is present as one molecule as well in kaolinite; SiO_2 is present in all, but in different ratios to the other oxides, making up all of the quartz, being sixfold as much as K_2O, Na_2O, and Al_2O_3 in orthoclase and albite, and twice as much as CaO or Al_2O_3 in anorthite and kaolinite; H_2O is only present in kaolinite, where it is equal to the SiO_2 and twice as much as the Al_2O_3, and it may also be present, uncombined chemically, as moisture.

The matter now resolves itself into algebra of a very simple sort. For convenience and simplicity, at the start we shall assume that orthoclase ($K_2O.Al_2O_3.6SiO_2$) is the only feldspar present. To the (molecular) K_2O present we assign an equal (molecular) amount of Al_2O_3,[1] and six times the (molecular) amount of SiO_2, to form the orthoclase molecule. As free alumina does not exist in clays, the rest of the Al_2O_3 must go into kaolinite; so of the SiO_2 we set aside for this mineral molecule an amount out of what

[1] If we take albite and anorthite into account, we should assign, of the Al_2O_3, amounts equal to those of Na_2O and CaO to form these minerals, respectively, and of the SiO_2 six times that of the Na_2O to form albite and twice that of the CaO to form anorthite.

is left over from the feldspar equal to twice the amount of the residual Al_2O_3; we set aside also an equivalent amount of H_2O. This uses up all of the K_2O and Al_2O_3 present, and there is left excess SiO_2 and probably H_2O. The SiO_2 is the quartz molecule, and the excess H_2O represents moisture.

We now have the molecular amounts of the orthoclase, kaolinite, and quartz, and to reduce these to mineral percentages we simply have to reverse the operation of obtaining the molecules; that is, we multiply the molecular amounts by the molecular weights. This can be done by multiplying the molecular amount of each assigned portion of each individual oxide by its proper molecular weight; but it is easier and more expeditious to multiply the molecular weight of each mineral by the figure that expresses the molecular amount of this mineral. This figure is simply the molecular number of that constituent oxide of which only one molecule is present in the mineral. Thus, for kaolinite it would be the molecular amount of Al_2O_3 present in kaolinite, for orthoclase the molecular amount of either K_2O or Al_2O_3 present in orthoclase, and for quartz the molecular amount of the residual SiO_2. The results of these multiplications will be the mineral or "modal" percentages by weight of the various minerals present, and their sum is the mineral or "modal" composition of the clay.

This process may strike one as very complex and laborious, but it is, in reality, very simple and rapid, particularly after one has had a little practice. Let us take an example, a very simple one to begin with. A clay has the following chemical composition:

	Per cent.	Mol. No.
SiO_2	63.36	$\frac{63.36}{60} = 1.056$
Al_2O_3	24.28	$\frac{24.28}{102} = 0.238$
K_2O	3.29	$\frac{3.29}{94} = 0.035$
H_2O	9.07	$\frac{9.07}{18} = 0.504$
	100.00	

To calculate the orthoclase first, we multiply its molecular weight, 556, by the unit molecular amount present, 0.035; we have then, $556 \times 0.035 = 19.46 = Or$. The amount of Al_2O_3 required for orthoclase, 0.035, is subtracted from the total amount, 0.238, and the remainder, 0.203, serves as a measure for the kaolinite. So we multiply the molecular weight of kaolinite, 258, by the unit molecular amount: $258 \times 0.203 = 52.37 = Kl$, the percentage of kaolinite. All the K_2O and the Al_2O_3 have thus been assigned; the only mineral present now remaining to be calculated is quartz. We therefore subtract from the total molecular amount of SiO_2, 1.056, the amount used for orthoclase ($6 \times 0.035 = 0.210$) and that used for kaolinite ($2 \times 0.203 = 0.406$), together 0.616; and multiply the remainder, 0.440, by the molecular weight of quartz, 60. We thus have, $60 \times 0.440 = 26.40 = Q$, as the percentage of quartz. We have now only to consider the water. From the total molecular amount, 0.504, we subtract that which was used for kaolinite, $2 \times 0.203 = 0.406$, and multiply the remainder by the molecular weight of water, 18; the product is the percentage of moisture; thus, $18 \times 0.098 = 1.76 = Aq$. The mineral composition of the clay is, therefore, as follows:

Kl (kaolinite)	52.37
Or (orthoclase)	19.46
Q (quartz)	26.40
Aq (moisture)	1.76
	99.99

This represents the actual mineral composition of such a simple mixture with great exactness, provided of course, the chemical analysis on which it is based is correct; and such a mode is far more accurate and trustworthy than the results of any "rational" analysis made by the often recommended methods.

Let us take a more complex case, in which lime, soda, and ferric oxide are present in addition to the oxides present in the clay just calculated. Suppose that the composition by chemical analysis is:

	Per cent.	Mol. No.
SiO_2	63.36	1.056
Al_2O_3	23.76	0.233
Fe_2O_3	0.52	0.003
CaO	0.69	0.012
Na_2O	0.97	0.016
K_2O	1.63	0.017
H_2O	9.07	0.504
	100.00	

Here (assuming the absence of calcite) the lime (CaO) forms part of residual anorthite, $CaO.Al_2O_3.2SiO_2$, or possibly one of the calcic zeolites, which contain hydrated anorthite molecules. The soda (Na_2O) forms part of residual albite, $Na_2O.Al_2O_3.6SiO_2$, or possibly may form part of a sodic zeolite, in which hydrated albite or nephelite molecules are present,[1] the difference in silica content of which is of negligible moment. The ferric oxide may be assumed to be present as limonite (Lm), $Fe_2O_3.H_2O$,[2] the most common of the hydrated ferric oxides. The calculation is briefly done thus: after calculating the orthoclase, albite, and anorthite, the remaining alumina serves as a basis for calculating the kaolinite; the ferric oxide is the basis for calculating limonite; the silica remaining from the orthoclase, albite, anorthite, and kaolinite is the quartz, and the water remaining from kaolinite and limonite is moisture.

The slight deficiency in the summation is caused by the errors in not carrying out the calculations of the molecular numbers beyond the third decimal place, which in this case all tend to low results.

The calculation is represented thus:

[1] The presence of zeolites in clays, and their composition, cannot be discussed here; the lime and soda are present to so small an amount in usable clays that errors in the assumptions above are of negligible consequence.

[2] This is the formula for limonite as determined by E. Posnjak in an investigation of the hydrated oxides of iron carried out in the Carnegie Geophysical Laboratory, and to be published in the *American Journal of Science*.

Or	= 556 × 0.017	=	9.45
Ab	= 524 × 0.016	=	8.33
An	= 278 × 0.012	=	3.34
Kl	= 258 × 0.188[1]	=	48.50
Lm	= 178 × 0.003	=	0.53
Q	= 60 × 0.458[2]	=	27.48
Aq	= 18 × 0.125[3]	=	2.25
			99.88

If carbon dioxide (mol. wt. = 44) is found, it is assumed to be present in calcite (mol. wt. = 100); an equivalent amount of CaO is allotted to the CO_2 for calcite, the calcite is then reckoned by multiplying the molecular number of CO_2 by 100, and the rest of the lime is calculated to anorthite, as above.

Magnesia, MgO (mol. wt. = 40), may be assumed to be present in chlorite, a complex mineral, or rather mineral group, into which biotite usually alters; but it is best treated on the assumption that it enters serpentine, $3MgO.2SiO_2.2H_2O$ (mol. wt. = 276), the unit of calculation being one-third of the molecular number of the MgO present. Any error involved will be quite negligible.

The micas remain to be considered. Of these, biotite may be disregarded, partly because it is seldom present in more than extremely small amounts in usable clays, and also because, if it is desired to take it into consideration, its amount may, without any practically appreciable error, be assumed to be that of serpentine.

Muscovite is more commonly present, though in small amount. Its composition may be considered as a mixture of orthoclase, alumina and water, so that the percentage amount in a clay cannot be calculated from a chemical analysis alone if orthoclase and kaolinite are present also. However, as orthoclase and muscovite have about the same fluxing effect,[4] it will serve all practical purposes to calculate the potash of any muscovite

[1] 0.188 = 0.233 — (0.017 + 0.016 + 0.012).

[2] 0.458 = 1.056 — [(6 × 0.017) + (6 × 0.016) + (2 × 0.012) + (2 × 0.188)].

[3] 0.125 = 0.504 — [(2 × 0.188) + 0.003].

[4] Ries, "Clays," p. 102.

present as orthoclase and the alumina as kaolinite, some of the excess silica being taken for this.

It will be obvious that the separate determination of potash and soda is necessary for the proper calculation of the feldspars. In many analyses of clays, these are not separately determined, but are reported as "alkalies," that is, as the oxides. In the course of analysis the alkalies are weighed together as chlorides, and to calculate these as oxides either the potash and the soda must be determined separately or the chlorides are assumed to be made up in arbitrary (and unknown) proportions of potash and soda. The latter procedure is highly unscientific and unsatisfactory, and it should not be countenanced by workers in ceramics.[1]

In order to show the actual results of such calculations as compared with the compositions deduced from "rational" analyses, the calculated norms of several analyses of clays tabulated by Ries,[2] who also gives their "rational" analyses, are presented in Table I. As, in these analyses, potash and soda have not been determined separately, the calculations have been made on the two assumptions: (1) that the "alkali" is all potash, the alkali feldspar being orthoclase; (2) that it is all soda, the alkali feldspar being then albite. The small amounts of magnesia are disregarded in these calculations.

Several points are clearly evident from a study of these calculated modes as compared with the "rational" analyses; and it is to be remembered that these are only a few of those that have been calculated. It is to be assumed that the chemical analyses are fairly correct, in view of the simplicity of the determinations of the few constituents present.

The first point is that the "rational" analyses do not generally agree with the mode; indeed, some of them are hopelessly and almost ludicrously incongruous. In I, the R. A. ("rational" analysis) while showing about the correct amount of kaolin, gives several times the amount of "feldspar" that could possibly be present on the basis of the alkalies. On the other hand, the R. A. of II

[1] Cf. Washington, *Prof. Paper*, **99**, p. 15; Mellor, "Quant. Anal.," p. 222.
[2] H. Ries, "Clays," **1914**, p. 67.

TABLE I.

Analyses.

	I.		II.		III.		VII.		VIII.		IX.		X.	
	Per cent.	Mol. No.	Per cent.	Mol. No.	Per cent.	Mol. No.	Per cent.	Mol. No.	Per cent.	Mol. No.	Per cent.	Mol. No.	Per cent.	Mol. No.
SiO_2....	62.40	1.040	62.52	1.042	63.17	1.053	53.10	0.885	47.60	0.793	46.61	0.777	58.39	0.973
Al_2O_3...	26.51	0.260	25.57	0.251	25.09	0.246	33.06	0.324	34.00	0.333	36.47	0.359	27.52	0.270
Fe_2O_3...	1.14	0.007	0.92	0.006	0.64	0.004	1.18	0.008	1.30	0.008	2.81	0.018	0.36	0.002
MgO....	0.01	..	0.10	..	0.26	..	0.08	..	0.50	..	None	..	0.41	..
CaO....	0.57	0.010	0.65	0.012	0.35	0.006	0.38	0.007	Trace	..	0.14	0.003	None	..
Alkalies.	0.98	0.010	1.04	0.011	0.80	0.009	0.83	0.009	3.00	0.032	1.44	0.015	4.29	0.046
		0.016		0.017		0.013		0.013		0.048		0.023		0.069
H_2O....	8.80	0.490	9.27	0.515	9.70	0.539	11.32	0.629	13.60	0.756	12.80	0.711	7.19	0.400
	100.41		100.07		100.01		99.95		100.00		100.27		99.68	

Modes.

	Or.	Ab.	Or.	Ab.	Or.	Ab.	Or.	Ab.	Or.	Ab.	Or.	Ab.	Or.	Ab.
Q.....	28.80	27.96	29.76	28.32	31.50	30.54	12.06	11.10	0.00	—3.90	0.06	—1.86	14.94	9.42
Or.....	5.56	...	6.12	..	5.00	..	5.00	..	17.79	..	8.34	..	25.58	..
Ab.....	..	8.38	..	8.91	..	6.81	..	6.81	..	25.15	..	12.05	..	36.15

An.....	2.74	2.74	3.34	3.34	1.67	1.67	1.95	1.95	None	None	0.83	0.83	7.51	7.51
Kl.....	61.92	60.37	58.82	57.28	59.60	58.57	79.46	78.43	77.66	73.53	87.73	85.66	50.83	44.89
Lm.....	1.26	1.26	1.08	1.08	0.72	0.72	1.44	1.44	1.44	1.44	3.24	3.24	0.37	0.37
Aq.....	0.05	0.27	0.95	0.77	1.28	1.45	0.09	0.23	2.63	3.20	None	0.52	0.07	0.90
	100.33	101.00	100.07	99.70	99.77	99.76	100.00	99.96	99.52	102.32	100.20	102.30	99.30	99.24
										3.90		1.86		
										98.42		100.44		

Rational Analyses.

Clay substance	66.33	72.05	67.82	83.39	88.34	96.08	55.88
Quartz.....	15.61	27.78	30.93	14.99	8.95	1.93	5.95
Feldspar.....	18.91	0.10	1.25	1.57	2.73	1.99	38.17

I. Crude kaolin, Webster, N. C.
II. Slip-clay, Lothain, Saxony.
III. Slip-clay, Kaschkau, Germany.
VII. Kaolin, West Mills, N. C.
IX. Fire-clay, Bautzen, Germany.
X. Kaolin, Limoges, France.
VIII. White clay, Wiesau, Germany.

yields only 0.10 of feldspar although 1.04 per cent of alkalies are present, which demand either about 6 per cent of orthoclase or 9 of albite. The presence of 3.34 of anorthite is also to be considered. Nos. III, VII, VIII, and IX show the same feature. It cannot be objected that the difference is due to adsorbed alkali—since the amount of this would be quite insufficient to account for the discrepancy. No. X shows in its R. A. somewhat high feldspar, but rather of the correct order of magnitude, while the quartz is somewhat too low, and the kaolin too high. It is evident from these results that there is no constant direction of error in the figures of a rational analysis as regards any of the constituents, and that they are therefore quite unreliable and unworthy of use.

The second point brought out is that, in most of the R. A.'s, a very considerable proportion of the feldspar has been dissolved by the treatment with acid and has been reckoned as "Clay Substance." This is clear in II, III, VII, VIII, and IX.

It would also appear that the feldspar is generally almost purely potassic, at least when little lime is present, though it is also evident that the anorthite molecule is present in rather notable amounts in the feldspars of some clays. It is to be remembered that the isomorphous presence of anorthite in the plagioclases increases their solubility in acids. The fact that the feldspars in the clays represented by the above analyses are chiefly orthoclase is indicated in the modal analyses VIII and IX, which show a deficiency in silica. This deficiency is caused by the lesser molecular weight of soda, which takes more silica to form albite than does potash to form orthoclase.

It need only be said, in conclusion, that the utter unreliability of the rational analysis may be considered to be established, and that the term "rational," as applied to this method of determining the mineral composition or mode, is not only a misnomer but contrary to truth.

Attention must also be again called to the fact that, while the suggested method for calculating the mode may appear to be complex and "theoretical," yet it really is simple and expresses very accurately the actual mineral composition. This is so, because of the extremely simple character of the mineral mixture,

the simplicity of the mineral molecules, the impossibility of the entrance of other mineral molecules into the problem to any disturbing extent, and finally the absence of the necessity for considering the molecules of the minerals present as made up of other assumed ones, a difficulty that is met with in the calculation of the modes of igneous rocks when such minerals as aluminous pyroxenes, amphiboles, and micas are present.

This method of calculation is not the only one that might be adopted for arriving at the same results. Thus, without reducing the percentages to molecular numbers, the proportions of kaolin, feldspars, and quartz can be calculated from the percentages directly. Thus the percentage of orthoclase can be calculated from that of potash by multiplying this by 1.085 for the alumina and by 3.832 for the silica, and adding the three figures. The amount of alumina so obtained is to be deducted from the percentage of alumina shown by the analysis, in order to get the basis for kaolin, and the amount of silica is to be deducted from that of the analysis for the balance that goes to quartz and kaolin. The several factors necessary are obtained by calculating the ratios of the different constituents of each mineral to the unit constituent. This will yield the same results as the method described above, but will obviously call for much more numerous calculations. If tables are used the calculations involved in the proposed method are almost entirely done away with.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
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THE ACTION OF ACETIC ACID SOLUTIONS OF DIFFERENT STRENGTHS ON A SHEET STEEL ENAMEL.

By Leon J. Frost, Cleveland, Ohio.

For commercial reasons it became desirable to further prove that a 20 per cent acetic acid solution exerts the greatest corrosive action on the type of vitreous enamel generally used on kitchen utensils.

It occurred to us that it might be worth while to place the data resulting from our experiments on record, especially since it bears out so well the work of R. D. Landrum[1] on the same subject.

The procedure was to allow equal volumes of the various strengths of acid to act on the inside surface of small enameled dishes and to determine the loss in weight resulting from this action.

The dishes for this purpose were prepared by coating small basins with, first, a coat of ground and then a thin coat of white enamel. Over these was applied a good coating of the glaze to be tested. The molecular formula of this glaze was as follows:

$$\left.\begin{matrix} 0.885\ Na_2O \\ 0.085\ K_2O \\ 0.015\ CaO \\ 0.015\ MnO \end{matrix}\right\} 0.263\ Al_2O_3 \left\{\begin{matrix} 2.291\ SiO_2 \\ 0.452\ B_2O_3 \\ 0.701\ F_2 \end{matrix}\right.$$

The enamel frit was milled with 8 per cent of Vallender Clay and 1 per cent of magnesium sulphate. Each dish was marked so as to show the per cent of acid to be used in it and these markings were thoroughly burned in. Only perfect dishes were used so as to minimize any chance of error. The dishes were then carefully cleaned, dried on a hot plate and cooled in a desiccator, after which they were accurately weighed. The required amounts of distilled water and of technically pure acetic acid to make 25 cc. of solution in each case were added from burettes and the

[1] *Trans. Am. Ceram. Soc.*, **14**, 489 (1912).

solutions were taken to dryness on a hot plate, the temperature of which was maintained as uniform as possible. This temperature was such as to cause a slight boiling but not sufficient for any loss from spattering of the solution. Evaporation required about 25 minutes. As soon as the dishes were dry they were removed from the hot plate and, after cooling, were washed and scoured with a soft abrasive so as to remove all material loosened by the action but so as not to affect the enamel itself. After rinsing with distilled water, the dishes were again dried on the hot plate, cooled in the desiccator and weighed.

An attempt was made to keep the conditions of testing as constant as possible. In spite of this care some of the results showed inconsistencies which warranted their being omitted. These inconsistencies were due mostly to chipped rims and pin blisters.

The strengths of the acetic acid solutions used and resulting losses in weight are tabulated as follows:

Per cent acid.	Gr. loss.	Per cent acid.	Gr. loss.	Per cent acid.	Gr. loss.
1	0.0056	17	0.0140	50	0.0168
2	0.0108	18	0.0151	60	0.0117
3	0.0127	20	0.0160	70	0.0076
4	0.0140	21	0.0214	80	0.0026
5	0.0111	24	0.0223	90	0.0013
7	0.0088	27	0.0184	100	0.0003
9	0.0127	30	0.0132	...	..
15	0.0132	40	0.0179	...	..

The results are better shown by the curve (Fig. 1). This curve does not check exactly with those presented by Landrum but does show a very similar variation. The fact that there is a marked deflection, greatest at 30 per cent instead of at 25 per cent as reported by Landrum, might be explained by our having used the technically pure instead of the chemically pure acid. There is no apparent explanation for the irregularity between 2 per cent and 9 per cent but from the results of several duplicate trials we are convinced that this was not entirely the result of error.

It is evident from this curve that a 4 per cent to 5 per cent (by volume) solution of such an acetic acid as would probably be

used for testing at most cooking utensil plants is well up in strength as regards its action on the enamel surface. This per cent acid is also of about the same strength as the strongest vinegar—which is probably the most destructive agent to which an enameled

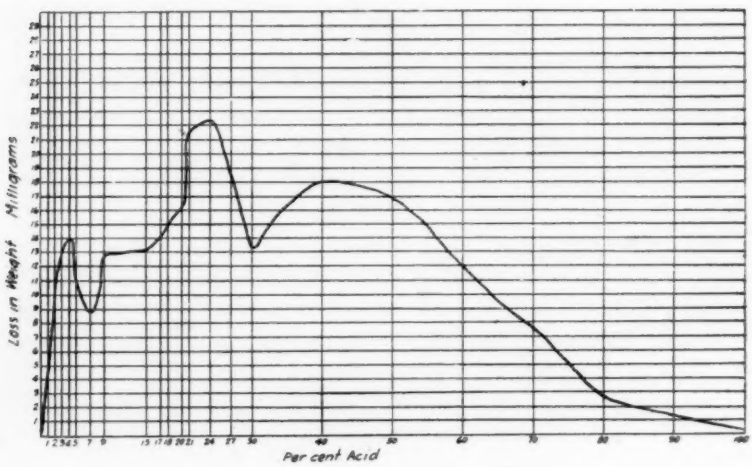

FIG. 1.

cooking utensil is subjected in ordinary use. The high point from 20 per cent to 25 per cent proves satisfactorily the point under investigation.

COMMUNICATED DISCUSSIONS.

J. B. SHAW: It seems to me that this work would have a little more value if the glaze selected for test had been of such a composition as to be less easily attacked by the acid. The glaze used is an alkaline boro-silicate, very high in fluorine, and therefore very soluble in water. While it is similar in its composition to enamels, it would make a very low grade enamel for even cooking utensils and could not be considered for use on ware requiring any special quality as regards acid resistance.

The data obtained seems to prove roughly the original assumption that about 20 to 25 per cent acid is most active on this enamel, but the method of making the test admits so many

possibilities of error that the value of the data is open to question. For instance:

(*a*) There is no doubt that the strength of the acid varies in every case throughout the period of evaporation.

(*b*) The rate of evaporation influences the results very materially.

(*c*) Scouring the surface with abrasives would surely introduce some error.

(*d*) Loss in weight would be proportional to the surface exposed. The same volume of acid does not necessarily act upon the same area of surface.

It is noted that care was taken to keep conditions constant, but after all precautions have been taken it seems likely that the possible error would exceed the difference between the results obtained with the 4 per cent and 24 per cent acid solutions.

No solvent except water can be maintained constant in a composition during the period of evaporation to dryness. Because of this fact it is impossible to obtain uniformly consistent results in such tests.

I believe a more reliable method of testing solubility—based upon the subjection of a given surface to a solution of constant strength at constant temperature for a short period of time—could be devised.

E. P. Poste: An interesting feature in connection with the action of acetic acid on enamels came to our attention in the case of a closed unit operating on strong acetic acid at elevated temperatures and under several pounds pressure. An enamel formula, which had been in successful operation in the canning industries under all possible conditions of acidity, and which had also been used on a large number of open units and stills for the stronger acetic acid solutions in various pharmaceutical and chemical manufacturing operations, was specified for this job. The enamel in question failed where the vapors had come in contact with the enamel—but was in perfect condition where it had only been in contact with the liquid. In other words, the action of the acid vapors at elevated temperatures and under pressure was much more severe than that of the liquid. Similar observations have

been made in connection with hydrochloric acid. The volatility of these acids would seem to account for the phenomenon. It has no direct bearing on the action of acetic acid on cooking ware unless it be the possible action of vapors in a covered kettle. However, the lack of pressures under these circumstances would seem to indicate that the case is not parallel.

B. A. RICE: Mr. Frost has very nicely accomplished his purpose and checked previous investigations. It is of interest to note that he secures the same peculiar dip in his curve—just beyond the high point—as did Landrum.[1] The fact that the percentage losses obtained by Mr. Frost are slightly higher is probably accounted for by the fact that he used technically pure instead of chemically pure acid.

Without a doubt the author has chosen the most desirable method of testing the acid resistance of his enamel, that is, by testing the enamel after it is burned on the ware. However, it would have been interesting to have tried checking this curve by subjecting the ground frit to the test outlined by Poste.[2] Poste compared his acetic acid curve with those obtained by Landrum,[1] but since he was using an enamel of fairly high acid resistance, the high points of the curve were not brought out as clearly as they might have been had a softer enamel, such as is used in cooking ware plants, been used. Should the curves resulting from the testing of an enamel by these two methods be of the same general nature—though the actual gravimetric values would differ—it is quite evident that testing the ground frit would be a much more simple laboratory method of controlling the acid resistance of enamels, once the acid resistance of a standard enamel had been established.

As pointed out by Staley in his discussion of Landrum's article, the method used by the author is accompanied by some very undesirable features: (1) The different rates of evaporation, (2) the variation in the strength of solution—due to evaporation, (3) the constantly changing surface area exposed to the action of the acid. These might be eliminated by simply covering the

[1] *Trans. Am. Ceram. Soc.*, **13**, 494 (1911).
[2] *Ibid.*, **18**, 137 (1915).

dish with a watch glass and heating at a constant temperature below boiling for a certain length of time. This would allow two means of obtaining results—(1) the loss in weight of the dish, (2) the determination of the amount of dissolved material in the solution. The objection to this method would be the possible effect of the vapors on the enamel. This factor could be reduced to a minimum by filling the dish as full as possible.

It would also be interesting to see the tabulated results of the action of cold solutions on the same enamel after standing a certain length of time. Would a curve plotted from these results be similar to the one obtained with the hot acids?

It might be well to mention also that tartaric acid from cooking grapes, as in making grape marmalade, has a much more severe action on enamels than acetic acid.

The foregoing points will undoubtedly be covered thoroughly by the Sub-committee on Enamels of the Committee on Standards, American Ceramic Society, in its work on the standardization of enameled ware. Some very useful and instructive data will be available for publication by this Committee in the near future.

R. R. DANIELSON: I believe a statement of the number of trials used in each case would throw some light on the irregularities in the data presented by the author. A limited number of trials would undoubtedly cause irregularities—due possibly to such mechanical defects in the preparation of the samples as overburning, etc., which might not be apparent under casual observation.

I would suggest tests in the interval between 18 and 30 per cent and with variations of 1 per cent. Having this data, it is possible that the author's data might approach more closely the results secured by Landrum and would at the same time make his results more conclusive.

L. J. FROST: Mr. Shaw brings up some very interesting points. It is true that the glaze used was somewhat less acid resistant than the ordinary cooking utensil enamel, but it was chosen purposely on that occount so that the variations in the curve would be contrasted the more strongly.

In view of the fact that the same volume of solution was used

in dishes having the same dimensions and the same number of coats of enamel in each case, and that the rate of evaporation was practically the same with every dish tested, I feel that the amount of error introduced here was too small to seriously alter the results. Admitting that the strength of the acid solution does vary in every case, throughout the period of evaporation, it still seems that the action of the acid solutions of different strengths should have the same relation as long as the rate of evaporation does remain practically constant.

A simple method of testing, and one which would give more accurate results in commercial investigations of this nature, would be welcomed by the writer.

The suggestions of Mr. Rice that the curve given be checked by the use of cold solutions, only, for a sufficient period of time, and that this same enamel frit be also tested by the method used by Poste, are very good. The objection to the latter method for control purposes, however, is due to the fact that conditions in sheet steel enameling often necessitate mill additions of so varied a nature as to materially affect the acid resistance of the completed enamel. If the frit alone is tested, this possible variation is lost sight of.

The proposed improvements on the above tests are very worthy of investigation.

Mr. Danielson is correct in his belief that over-burning and other unavoidable conditions of manufacture do cause some irregularity in the results. In running a large number of tests, always with a 20 per cent solution and on the same enamel—which was much more acid resistant than the one given above—I have found 1 to 1.5 mg. loss to be the usual limits of variation. However, when testing two dishes coated from the same lot of enamel and burned at the same time, the results have checked exactly.

NOTE ON COLORS PRODUCED BY THE USE OF SOLUBLE METALLIC SALTS.

By Paul G. Larkin, Denver, Colorado.

Introduction.

For a number of years soluble metallic salts have been used in under-glaze color work. In 1910 experimental work developed that this method could be employed as a means of producing over-glaze color in polychrome terra cotta.

The usual methods of production of polychrome terra cotta involve the painting of colored glazes in the required areas with a camel's hair brush—or the use of shields or stencils to protect certain areas while a colored glaze is sprayed on other portions of the piece. These, and numerous other devices, are objectionable on account of the time consumed, the results obtained, and the uncertainty of the whole process. They are presumably too well known to require further discussion here.

The use of soluble metallic salts offers a partial solution of this problem. By this method the pieces requiring the specified colors are first sprayed with the glaze or slip to be used as a base—the soluble salt solution then being applied to the indicated areas by hand painting. A steady hand and a little judgment are the only requisites for the application. The simplicity of the operation and the rapidity with which the over-glaze solution is applied are decidedly in favor of this method—when compared with the usual operation. To facilitate the application, it is necessary to have a base glaze or slip with an even, smooth surface such as would be obtained at the spray table from a water-glazed piece. A rough or granular-surfaced piece is painted with difficulty and seldom permits of smooth, clean-cut lines. The glaze used should contain a certain amount of gum arabic, molasses, or a similar substance affording a bond of sufficient strength to enable the piece to withstand the handling required during the color application. Generally, standard finish terra cotta is covered with an engobe having a clay content sufficiently high to

give a bond which, with reasonable care, will undergo handling without chipping or rubbing off.

For the past $2^1/_2$ years this scheme has been used at our plant with good results. It has been used with matt, full-glaze, and standard finish terra cotta. The application on matt glazes, as a whole, gives the best results and the greatest color range—producing colors of cleaner shade than those possessed by either the full-glazed or standard finish ware.

The process is best adapted to color designs in which the area to be treated is comparatively small—particularly if the solution is to be applied by a brush. Uniformity of color may be obtained over large areas by spraying the solution, but this is successful only in the case of flat surfaced pieces—where it would probably be as economical to supply the required color by a glaze containing metallic oxides.

In one instance, a color scheme required twelve colors. The smaller color areas, ten in number, were obtained through the use of soluble salts, while the larger remaining color areas were produced by spraying colored glazes. The colors produced are not entirely free from variation and in a large unbroken area are prone to show unevenness. Nevertheless, the color is generally applied to ornamental pieces and the lights and shadows offset the variations. It is on this last-named type of work that the application of colored glazes gives the greatest difficulty, and, despite the variation obtained through the use of soluble salts, the method produces better results in a shorter time. Ornamental insert pieces, in one, two, or more colors and commercial features, such as name panels and trade marks in color, are easily and quickly manufactured.

Application.

In the application of these color solutions, it is not advisable to use a full brush of the solution as it spreads rapidly and, if applied in this fashion at a point where a clean line is required, gives a blurred outline. The better practice is to use a full brush in the center of a color area and, as the supply of the solution in the brush is depleted, to work smoothly and without undue spreading to the outer edges in order to secure a cleaner line.

One application is sufficient if the surface is thoroughly covered. Solutions that produce a streaked appearance after firing are not improved by a second coating applied in a direction at right angles to the first application. The effect is merely changed from a striped to a checkered one.

Preparation.

The following represents the method employed in the preparation of the different blends:

We arbitrarily adopted a standard solvent which consisted of 75 per cent water (not distilled) and 25 per cent glycerine (by volume). Standard solutions of nine soluble metallic salts in this standard solvent were made by adding an arbitrarily adopted weight of each salt to the solvent. A supply of these solutions was kept on hand, in glass-stoppered bottles.

To each standard solution was added 10 cc. of wood alcohol to every 100 cc. solution (or roughly 10 per cent of wood alcohol). The different standard solutions were blended by the use of burettes on a volumetric basis. Aniline dye in solution in alcohol was added to the blends in the same proportion as added to the standard solutions, *i. e.*, 10 cc. aniline-alcohol solution to each 100 cc. of blended solution. For this purpose red, green, blue, violet, yellow, brown and black aniline dyes were used in order to lend color to the solutions and thus aid in the application. This method offers a simple basis for experimental work but does not admit of ready comparison.

FORMULAS—Glazes and Engobe.

The following formulas represent the two types of glazes on which the soluble over-glaze colors were applied:

FULL GLAZE (FRITTED) (CONES 2–3).

$$\left.\begin{matrix} 0.25\ K_2O \\ 0.25\ PbO \\ 0.30\ CaO \\ 0.20\ ZnO \end{matrix}\right\} 0.325\ Al_2O_3 \left\{ 2.20\ SiO_2 \right.$$

+ 6 per cent SnO_2
+ 0.2 per cent uranium oxide

MATT GLAZE (RAW TYPE) (CONES 2–3).

0.060 KNaO 0.073 K_2O 0.032 Na_2O 0.004 MgO 0.413 CaO 0.418 ZnO	0.25 Al_2O_3	1.7 SiO_2

The engobe used as the base for the standard finish terra cotta samples had a composition as follows:

STANDARD FINISH ENGOBE (CONES 2–3).

Batch weight. Per cent.	Frit. Per cent.
28.0 Feldspar	75.0 Red lead
23.0 Flint	25.0 Flint
10.0 Cornish stone	
4.5 Frit	100.0
21.0 Ball clay	(Melted down in a frit kiln.)
12.0 Georgia kaolin	
1.5 Borax	
100.0	

Standard Solutions.

The standard solutions used were prepared as follows from technically pure salts:

No. 1. 100 cc. solvent.
50 g. ferric nitrate.
10 per cent wood alcohol (by volume).

No. 2. 100 cc. solvent.
50 g. cobalt chloride.
10 per cent wood alcohol. "

No. 3. 100 cc. solvent.
50 g. uranium nitrate.
10 per cent wood alcohol. "

No. 4. 100 cc. solvent.
50 g. manganese nitrate.
10 per cent wood alcohol. "

No. 5.[1] 100 cc. solvent.
25 g. chromium nitrate.
2 g. zinc chloride.
10 per cent wood alcohol (by volume).

No. 6. 100 cc. solvent.
50 g. nickel nitrate.
10 per cent wood alcohol. "

No. 7. 100 cc. solvent.
50 g. copper nitrate.
10 per cent wood alcohol. "

No. 8. 100 cc. solvent.
50 g. tin chloride.
10 per cent wood alcohol. "

No. 9. 100 cc. solvent.
50 g. zinc chloride.
10 per cent wood alcohol. "

[1] We attempted to use larger amounts of the chromium nitrate in No. 5 but had difficulty in getting it into solution. The difficulty was due to the deterioration of the salt. Zinc chloride was used because a salmon color was desired.

BLENDS FOR GREEN COLORS ON ZINC BEARING GLAZES.

Solution.	No.	G-1. cc.	G-2. cc.	G-3. cc.	G-4. cc.	G-5. cc.	G-6. cc.	G-7. cc.	G-8. cc.	G-9. cc.	G-10. cc.	G-11. cc.
Ferric nitrate.....	1					4.5		2.5	8.0	5	4	3
Cobalt chloride...	2	25	20	10	16	17.0	3.0	2.5	2.0	4	5	6
Uranium nitrate..	3	25	22	20	20	19.0	12.5	10.0	10.0	8	8	8
Nickel nitrate....	6			10	5	4.5	9.5	10.0	5.0	8	8	8
Copper nitrate....	7			10								
Manganese nitrate	4		8		9	5.0						
Solvent.....	..						25	25	25	25	25	25

To each blend, 5 cc. of aniline dye in alcohol solution was added.

Each individual blend in the above table produced a green on matt glazes.

Blends Nos. G-4 and G-5 were the best greens obtained and were blue-green on matt glazes and blue on full or bright glazes with a slight green cast. Blue on standard finish ware.

Attempts were made to produce a green, using a combination of solutions No. 7 (copper nitrate), No. 8 (tin chloride), and No. 9 (zinc chloride). The results obtained on matt and full glazes

were not satisfactory. The color in nearly every case varied greatly—blotched in one part with a dark metallic green-black and a light dirty green in another part. Presumably, the base glaze used was not adapted to the production of a copper-green.

Incidentally, green colors were obtained through the use of uranium, but were not stable or dependable and were difficult to combine in harmony with other colors.

BLUE COLORS.

The lighter blues were produced by blending the standard solution of cobalt (solution No. 2) with standard solutions of manganese (No. 4), nickel (No. 6), and iron (No. 1), in varying proportions and in some cases by diluting the cobalt solution.

Blue No. 1:
- 7.5 cc. solution No. 6 (nickel nitrate).
- 2.5 cc. solution No. 1 (iron nitrate).
- 15.0 cc. solution No. 2 (cobalt chloride).
- 25.0 cc. solvent.
- 5.0 cc. aniline dye in alcohol.

Blue No. 2:
- 10 cc. solution No. 2 (cobalt chloride).
- 40 cc. solution No. 4 (manganese nitrate).
- 5 cc. aniline dye in alcohol.

Blue No. 3:
- 15 cc. solution No. 2 (cobalt chloride).
- 15 cc. solution No. 4 (manganese nitrate).
- 5 cc. solution No. 6 (nickel nitrate).
- 10 cc. solution No. 8 (tin chloride).
- 5 cc. solvent.
- 5 cc. aniline dye in alcohol.

Darker blues were produced by increasing the concentration of solution No. 2 (cobalt) by the further addition of cobalt chloride. A good dark blue had the following composition:

Blue No. 4:
- 40 cc. solution No. 2 (cobalt chloride).
- 8 cc. solution No. 4 (manganese nitrate).
- 2 cc. solution No. 1 (ferric nitrate).
- 5 g. cobalt chloride to blend.
- 5 cc. aniline dye in alcohol solution.

BROWN COLORS.

Standard solutions No. 1 (ferric nitrate), No. 3 (uranium nitrate), No. 4 (manganese nitrate), No. 6 (nickel nitrate), and No. 5 (chromium nitrate) were blended to produce brown colors. In general, the efforts were not successful—the colors being light and of a disagreeable shade. The best colors obtained consisted of blends as follows:

Brown No. 1:
19.0 cc. solution No. 4 (manganese nitrate).
7.5 cc. solution No. 1 (ferric nitrate).
11.5 cc. solution No. 6 (nickel nitrate).
12.0 cc. solution No. 5 (chromium nitrate).
5.0 cc. aniline dye in alcohol.

Brown No. 2:
10 cc. solution No. 5 (chromium nitrate).
18 cc. solution No. 4 (manganese nitrate).
2 cc. solution No. 3 (uranium nitrate).
5 cc. aniline dye in solution.

Brown No. 3:
30 cc. solution No. 1 (ferric nitrate).
10 cc. solution No. 6 (nickel nitrate).
10 cc. solution No. 4 (manganese nitrate).
5 cc. aniline dye in solution.

GRAY COLORS.

Several acceptable gray colors were produced in blends of solutions No. 1 (ferric nitrate), with a small quantity of solution No. 2 (cobalt chloride), No. 4 (manganese nitrate), and No. 6 (nickel nitrate). A tendency towards brown was noted in the application on full glazes or enamels. The variation is also marked.

Gray No. 1:
0.5 cc. No. 2 (cobalt chloride).
24.5 cc. No. 6 (nickel nitrate).
25.0 cc. No. 4 (manganese nitrate).
5.0 cc. aniline dye in alcohol.

Gray No. 3:
25.0 cc. No. 1 (ferric nitrate).
1.0 cc. No. 2 (cobalt chloride).
10.0 cc. No. 4 (manganese nitrate).
14.0 cc. No. 6 (nickel nitrate).
5.0 cc. aniline dye in alcohol.

YELLOW AND ORANGE COLORS.

The following blends represent the best colors obtained in combining solution No. 1 (ferric nitrate), solution No. 3 (uranium nitrate), solution No. 4 (manganese nitrate), and solution No. 5 (chromium nitrate). The colors are the results of experimental work in an attempt to produce yellow and orange colors

Y-1:
20 cc. No. 3 (uranium nitrate).
20 cc. No. 4 (manganese nitrate).
10 cc. No. 5 (chromium nitrate).
5 cc. aniline dye in alcohol.

Y-2:
10 cc. No. 1 solution (ferric nitrate).
9 cc. No. 3 solution (uranium nitrate).
16 cc. No. 4 solution (manganese nitrate)
15 cc. No. 5 solution (chromium nitrate).
5 cc. aniline dye in alcohol solution.

Y-3:
20 cc. No. 3 solution (uranium nitrate).
10 cc. No. 4 solution (manganese nitrate)
20 cc. No. 5 solution (chromium nitrate).
5 cc. aniline dye in alcohol solution.

Y-4:
5 cc. No. 1 solution (ferric nitrate).
20 cc. No. 3 solution (uranium nitrate).
20 cc. No. 5 solution (chromium nitrate).
5 cc. solvent.
5 cc. aniline dye in alcohol solution.

In attempting to introduce tin chloride as another component in Y-3, we obtained a brown-yellow of unpleasing shade.

The combination of an insoluble stain and a soluble salt gave better results in the production of yellow than the use of soluble salts alone.[1]

The following stain was mixed dry, calcined to cones 2–3, wet ground and sieved through a 150-mesh screen:

[1] *Trans. Am. Ceram. Soc.*, **19**, 653 (1917).

YELLOW STAIN.

White lead	22.6
Antimony oxide	20.2
China clay	15.8
Tin oxide	14.4
Calcium carbonate	11.2
Sodium nitrate	15.8
	100.0

After calcination at cones 2–3, the stain was dense, brown-yellow in color and rather hard.

This stain, suspended in standard solution No. 3 (uranium nitrate), produced a good orange-yellow color. The blend had the following composition:

Y-8:
35 cc. solution No. 3 (uranium nitrate).
30 g. yellow stain (150 mesh).
35 cc. solvent.
(No aniline dye required.)

Brush application shows variation. The color is good. Further attempts to improve the color by the introduction of 150-mesh rutile produced a lighter color with a brownish cast—not equal to Y-8 in strength. The Y-8 yellow is not as good on full glaze and on standard finish produces a faint yellow with no bond existing between the stain and the engobe.

Comparative Costs.

Some information may be gained on the cost of application of these soluble colors from the following data on a standard finish corner stone and a name panel:

A 40″ × 20″ rectangular corner stone with 28 V counter-sunk letters required $6^1/_2$ hours labor at $0.20 or $1.30 and materials costing $0.05, making an approximate total of $1.35.

The standard finish name panel contained 26 double V-countersunk letters with an approximate height of 8″. Two colors were applied, one to the outline V and a contrasting color to the deep V or body of the letter. The labor necessary to complete the

color application cost $1.60 and the materials added $0.22, making an approximate cost of $1.82.

Conclusion.

Naturally, there are certain colors whose pyro-chemical requirements cannot be satisfied by a solution application, but as far as this scheme can be carried out, it solves some very perplexing questions in polychrome terra cotta and doubtless can be employed in other fields.

As noted before, it is labor saving; it gives good results; clean lines are a matter of manipulation—except possibly on a full glaze where the color "creeps" during the firing operation (allowance should be made for this movement at the outside edges of the color area).

The addition of a lead frit or flux to a standard-finish engobe—making proper allowance for this addition so as not to bring the engobe to a glaze or semi-glaze condition—will improve to a marked degree the colors produced by the use of soluble salts on standard finish ware.

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NOTE ON THE SINTERING OF MAGNESIA.

By John B. Ferguson.

Prior to the war, pyrometer tubes, furnace tubes, and similar ware made from magnesia were imported from Germany. This source of supply is now cut off and a new source has not yet appeared. The sintering of chemically pure magnesia has been generally regarded as difficult, if not impossible, and the refractory articles referred to are, in fact, found to contain about two per cent. of forsterite ($2MgO.SiO_2$), together with about one per

Fig. 1.

cent. of other impurities (estimated roughly under the microscope), which act as a bond. It was thought of interest, therefore, to put upon record some observations on the sintering of silky, calcined, pure magnesia such as is used to insulate small electric furnaces.

In determining the melting points of cristobalite and tridymite,[1] a special electric resistance furnace of the cascade type was employed and the two heaters were insulated from each other by a

[1] J. B. Ferguson and H. E. Merwin, *Am. J. Sci.*, **46**, 417 (1918).

layer of silky, calcined, magnesia powder. The furnace was maintained for some hours at temperatures ranging from 1600° C to 1720° C and the magnesia packed so rapidly that it was necessary to add more of it every hour. Upon dismantling the furnace, the magnesia was found to have formed a dense cake surrounding the inner heater, as shown in the accompanying photograph. The cake had considerable mechanical strength. A sintering of this character has not before been observed in our furnaces, which operate usually below 1600° C.

A microscopic examination of this cake by Dr. H. E. Merwin, of this laboratory, did not disclose any binding material; only periclase (crystallized MgO) with less than one-half of one per cent of forsterite ($2MgO.SiO_2$) could be identified. The crystals of periclase were interwoven to a considerable extent.

The silica content of the cake was found by chemical analysis to be 0.44 per cent.

The manufacture of magnesia ware of sufficient mechanical strength without a binder would, therefore, appear feasible if the product were fired at temperatures between 1600° C and 1700° C for some hours.

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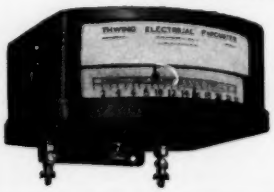

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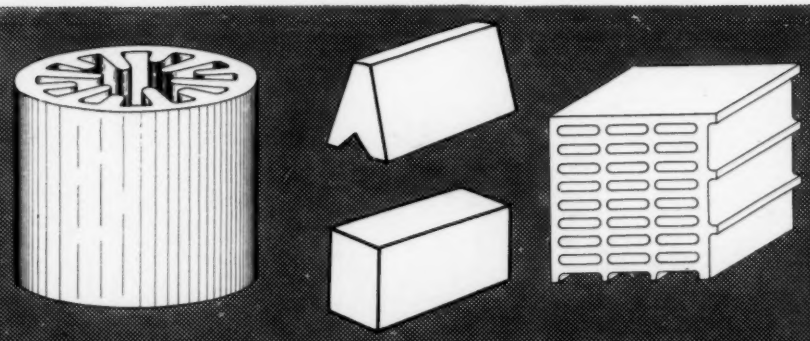

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